LABORATORY MANUAL OF GENERAL INORGANIC CHEMISTRY

SNEED AND KIRK

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PREFACE

This manual follows the order of chapters of Sneed's "General Inorganic Chemistry." More experiments are included than will normally be performed by any single class. Topics of a diversified nature have been selected to give the instructor a choice of material suitable to the purpose of his course.

Instead of adhering to the customary practice of directing students to use "a little of this" or "some of that" reagent, we have thought it in keeping with the tenets of an experimental science to give specific directions wherever possible. Volumes are expressed in milliliters instead of cubic centimeters, as recommended by the United States Bureau of Standards.

M. C. SNEED
R. E. KIRK

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PRELIMINARY LABORATORY DIRECTIONS

General Rules. *a.* The desk at which you work must be left clean at the end of each laboratory period.

b. Do not throw any solids into the sinks. Matches, filter paper, and the like should be thrown into receptacles provided for the disposal of solid waste material. If you spill substances on the top of your desk or down the front of it, clean the desk at once.

c. The apparatus in your desk is to be kept clean. Have a definite place for each article.

d. Close observation, cleanness, and neatness are essential to good chemical work.

Apparatus. A set of apparatus, covering most of the needs of the course, is lent to the student at the beginning of the term. Apparatus not found in the desk, but required in the course, may be borrowed from the storeroom. These articles should be returned dry and in good condition as soon as the experiment is completed.

Chemicals. *a.* Students will find on the reagent racks an alphabetically arranged set of chemicals covering most of the needs of the course. Chemicals not found on the shelves, but required in the work assigned, may be secured at the storeroom window.

b. Three rules governing the use of reagents are as follows: (1) Follow directions and be sure to use the quantities of reagents that are specified. (2) Avoid the use of an excess of any reagent when the amount is not given. Should more of a reagent be taken from a bottle than is required, the excess should not be put back into the reagent bottle. (3) Never put stoppers down on the desk. Avoid contamination of reagents by keeping each stopper clean and by quickly replacing it in the proper bottle.

Procedure for Laboratory Work. *a.* In order to secure reliable results in your laboratory work, you should acquire an orderly habit of procedure.

b. Read the assigned experiments before coming to the laboratory. Be sure that you understand what you are to do and the real purpose of each experiment. If necessary, consult your textbook, especially when directed to do so by the notation (Reference). Plan the work and note the apparatus and chemicals to be used. Write and balance equations for all chemical actions involved in the experiments. Work out all assigned problems. Base answers to all questions on careful observations and experiment.

c. When you enter the laboratory go at once to your desk and take out the equipment needed. Obtain the necessary chemicals and supplies. You will thus save yourself much time. Consult your laboratory instructor on doubtful points.

d. Follow directions carefully when performing an experiment. Be sure to use the exact amounts designated in the directions. Each person is to work independently save when express instructions to the contrary are given.

e. Each piece of apparatus is to be assembled very neatly and carefully. Assembled apparatus should face you squarely and should be firmly supported. The laboratory instructor's approval should be obtained before the apparatus is used. Any defects must be remedied before doing the experiment.

f. Before leaving the laboratory be sure that all equipment is clean and replaced in the desk. Turn off the water and the gas and clean the desk thoroughly.

g. Perform under a hood all experiments involving the production of noxious fumes and gases.

Directions for Laboratory Records. *a.* Give number and title of each experiment and the date when carried out. The record of the laboratory work should consist of brief statements of what you did, the results, and answers to questions. This record is to be made in ink. It must be clear and to the point. Neatness and legibility are essential.

b. Record your work and its results, and answers to all questions, in complete, well-arranged sentences. Be sure to say exactly what you mean. Form the sentence fully in your mind before writing it in your record book. Good English and correct spelling are required. Tabulate your results wherever possible.

c. Consult your instructor regarding points about which you are doubtful. When the work is completed and the record correctly made, present your notes for his approval.

d. If an experiment is repeated, the final report must contain both sets of data. Do not destroy the record of incorrect results, as you are likely to form a bad habit by so doing.

LABORATORY MANUAL OF GENERAL INORGANIC CHEMISTRY

CHAPTER I

INTRODUCTION

1. Bunsen Burner. Connect your Bunsen burner with the gas supply by means of a piece of rubber tubing (Fig. 1). Turn the ring at the bottom of the burner till the openings are closed and then light the burner. Note the color of the flame. Now turn the ring slowly until the flame becomes blue but burns quietly. What has caused this change? What is the name given to a similar mechanism on an automobile? Turn out the burner, attach the wing top, and light as before. Note the shape of the flame.

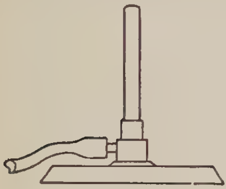

FIG. 1

2. Glass-Working. Cut several pieces of glass tubing, by making a scratch with one forward stroke of a triangular file on one side of the tubing. Hold the tube in both hands with the two thumb-nails opposite the scratch (Fig. 2). Pull the ends of the tubing toward you. You should obtain a clean break at a right angle to the length of the tube. Fire-polish several pieces of glass tubing, by holding their ends in the flame and constantly rotating the tubes until the flame turns yellow. This is an indication that the glass has begun to soften.

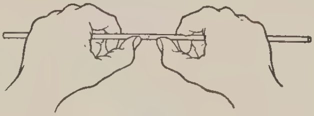

FIG. 2

Adjust the burner, with the wing top attached, until the flame is blue. Hold a piece of glass tubing in the flame so that about 2 in. of the middle portion will be heated (Fig. 3). Slowly rotate the tube between the thumbs and fingers so that it may be evenly heated. When the heated portion has become soft, remove the tube from the flame and bend it until the arms make a right angle. Work slowly, and make sure that the arms of the tube are in the same plane. The bend should be rounded and not angular or pinched. Why? Practice bending tubes until you can produce good bends.

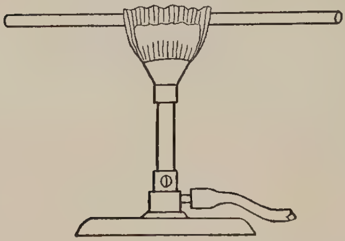
FIG. 3

Select a piece of tubing about 6 in. long. Bend it at right angles so that the arms will be of equal length. Fire-polish each end. Take another piece of tubing about 16 in. long. Bend it at right angles with the bend about 3 in. from one end. Fire-polish each end.

3. Assembling Apparatus. Take from your desk a Florence flask *A* and fit it with a two-hole rubber stopper (*storeroom*). Introduce a *moistened* thistle tube *B* into one of the holes of the stopper. Work it in slowly. Why? Introduce into the other hole the small right-angle tube. Connect the long tube with it by means of a short length of rubber tubing. The glass tubes should come close together within the rubber tubing. This piece of apparatus and all others must be set up carefully and neatly or it will have to be dismantled. Put the stopper in the flask, mount it on a ring *D* covered with wire gauze *E* and supported by a ring stand *C* (Fig. 4). Make the flask secure by means of a clamp *F*. Adjust the thistle tube until its end is about a quarter of an inch from the bottom of the flask.

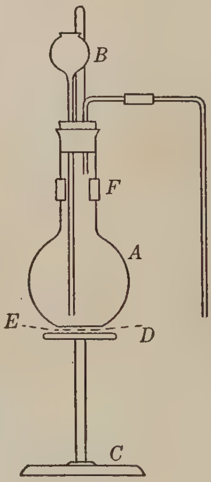

FIG. 4

4. Use of Apparatus as a Generator. Pour water through the thistle tube until its end is covered. Heat the water to boiling. Where does the steam come out? Why? Why should a thistle tube always dip into the liquid? Hold your thumb over the end of the delivery tube for a moment. What takes place in the thistle tube? Now let the delivery tube dip under the water in a beaker. Again boil the water in the flask. Then take away the flame. Repeat, holding the palm of the hand over the top of the thistle tube as the flask cools. Result? Give two ways in which the thistle tube acts as a safety device.

FIG. 5

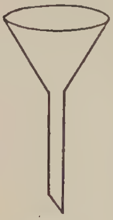
FIG. 6

5. Precipitation. Place 1 ml. of a solution of sodium chloride in a test tube (Fig. 5). Add to this 1 ml. of silver nitrate solution. The white solid which forms is said to be a precipitate. In this case it is insoluble silver chloride.

Allow the precipitated silver chloride to settle and then add a few more drops of the silver nitrate solution. If more silver chloride is seen to form, it is an indication that the initial precipitation was not complete. Allow the newly formed precipitate to settle and again add a few drops of silver nitrate solution. Repeat this operation until a failure to obtain more precipitate indicates that complete precipitation has occurred.

6. Filtration. The precipitate obtained in Experiment 5[1] may be separated from the liquid by pouring it upon filter paper supported on a glass funnel (Fig. 6). Fold a circular filter paper (Fig. 7) first into shape *A* and then into shape *B*. Open the paper into a cone having three thicknesses on one side and one thickness on the other. See if the cone will fit the funnel. If it does not, adjust it so that it will. Moisten the filter paper with distilled water so that it will adhere tightly to the funnel. Press the paper cautiously against the funnel to remove any air bubbles. After the funnel is placed in the funnel arm *A* (Fig. 8), the apparatus is ready for use.

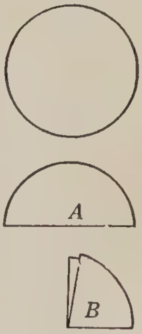

FIG. 7

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[1] References to experiments throughout the Manual will be by the numbers of the sections in which they are treated; thus, Experiment 125, *b*, will be found in section 125, part *b*.

The material from Experiment 5 is now poured down a glass rod *B* into the filter paper. A beaker *C* is placed below the funnel to catch the filtrate, that is, the liquid which passes through the filter.

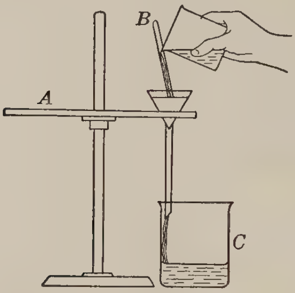

FIG. 8

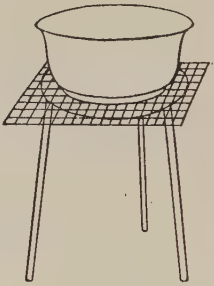

FIG. 9

7. Evaporation. Transfer the filtrate from the beaker to an evaporating dish placed on a wire gauze supported on a tripod (Fig. 9). Heat the dish carefully until crystals begin to appear. Then cover the dish with a watch glass (Fig. 10) and allow the contents to cool. Crystals of sodium nitrate will separate. Examine them carefully.

FIG. 10

8. Solution. Add 1 g. of common salt to 10 ml. of water in a test tube. Shake the contents (why?) until the salt dissolves. Repeat, using 10 g. of salt. Does all of it dissolve? What information do you obtain from this experiment other than that salt is soluble in water?

9. Collection of Gases. Gases which are not too soluble in water are often collected by the downward displacement of water. A pneumatic trough and wide-mouthed bottles filled with water are commonly used (Fig. 11). The bottles are filled with water, covered with a glass plate, and inverted. They are then placed on the shelf of the

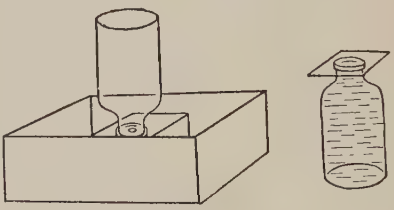

FIG. 11

pneumatic trough, and the glass plate removed. When the bottles are filled with the gas the glass plate is again inserted and the bottle removed.

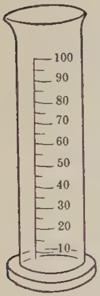

Fig. 12

Sometimes gases are collected by either the upward or downward displacement of air. Hydrogen is used in balloons. Should this gas be collected by downward or by upward displacement of air? Explain.

10. Measurement of Liquids. Liquids are measured by graduated cylinders (Fig. 12) or by burettes (Fig. 13). The first are used for ordinary purposes in your work. You should use the latter only when specified in the directions. In measuring the volume of water, for example, the lowest point of the curved surface of the liquid (meniscus) is the correct height for the reading.

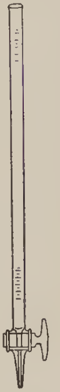

Fig. 13

11. Transference of Solids. Never transfer solids from reagent bottles directly into a test tube or evaporating dish. Use a piece of glazed paper and rotate the bottle slowly so that the solid may be caught on the paper in small amount. When the material is to be placed in a test tube, it is always best to crease the paper lengthwise before using.

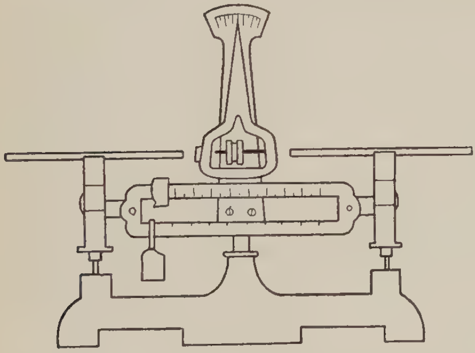

Fig. 14

12. Weighing. Weighing may be done on the platform balance (Fig. 14), on the horn-pan balance (Fig. 15), or on the analytical balance (Fig. 16). The last is used for accurate weighings. The horn-pan balance is more exact than the platform balance. No matter which of the foregoing is to be used the following directions should be observed:

See that the balances are clean.

Place material to be weighed on the left side and weights on the right.

Pieces of certain metals such as zinc and copper may be placed directly upon the platform or pan. Other substances must be weighed in glass, porcelain, or metal containers.

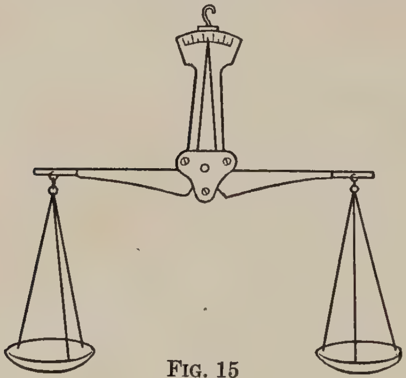

FIG. 15

Place the objects to be weighed and the larger weights in the middle of the platform or pan.

In weighing, the character of the substance will often determine the nature of the container in which it is to be weighed. If you are in doubt as to the kind of container, ask your instructor.

a. Platform Balance. The process of weighing on a platform balance is as follows: Obtain the weight of, or counterbalance, the container. Remember to put the substance on the left. On the right, place weights until the pointer swings an equal number of spaces on each side of the middle mark. Weighing objects of 5 g. or less may be done by moving the rider, on the graduated beam in front of the balance, to the proper position.

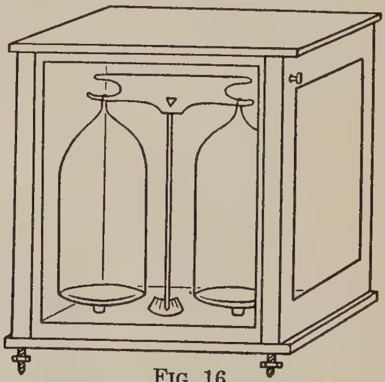

FIG. 16

b. Horn-pan Balance. The method of weighing on a horn-pan balance is essentially the same as that of the platform balance.

c. Analytical Balance. The following directions and precautions should be observed in the use of this balance:

Sit directly in front of the center of the balance.

See that the balance is level.

Release and arrest the pans. Always release the pans before releasing the beam.

Release the beam with a slow and steady movement of the screw or lever.

See if the pointer swings an equal number of spaces on each side of the middle line. If it does, proceed with your work. If it does not, note the exact position at each end of its swing and record these readings in your notebook. Arrest the beam as the pointer reaches the middle of the scale. Make another record of the swing of the pointer. By taking the average as illustrated in the following example, find the zero point:

Reading on Left	Reading on Right
5.6	8.2
4.2	6.8
Average 4.9	7.5

$$\text{Zero point} = \frac{7.5 - 4.9}{2} = 1.3 \text{ to right}$$

The pointer should swing through four or five divisions on each side of the middle point.

If the beam does not commence to swing as soon as it is released, set it in motion by wafting air over one of the pans with the hand, or by again raising and releasing the beam.

See that the rider is not so near as to be hit by the swinging beam.

Handle weights only with forceps.

The temperature of objects to be weighed should be that of the room.

Arrest the beam, place the object on the left pan, and the weight judged to be correct on the right pan. Follow a system in the selection of weights. Now release the beam carefully and note the movement of the pointer. After you have placed the proper weights on the pan, you are ready to use the rider.

Always close the balance case before starting to use the rider. The rider should be moved until the swing of the pointer is the same distance on each side of the zero point.

Record the result of your weighing at once in the proper place in your notebook. Check the weights as you remove them from the pan. One of the most common sources of error among beginners is the incorrect reading and recording of weights. When in doubt, ask the aid of your instructor.

Carefully weigh a watch glass. Record its weight and keep it as a container for some of the substances to be weighed later.

13. Wash Bottle. Construct a wash bottle, using Fig. 17 as a model. Use a two-hole rubber stopper. After you have completed your bottle, submit it to your instructor for his approval or rejection. It is provided with a nozzle so attached as to be movable. The instructor will demonstrate to the class the proper way to use this bottle in washing precipitates. When five or more milliliters of water are needed, it is customary to pour it out through the mouthpiece. *Use distilled water only.*

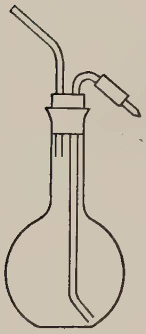

FIG. 17

14. Chemical and Physical Changes. *a.* Hold a piece of glass rod in the Bunsen flame. Wherein is it changed? Remove it from the flame. Is the change permanent? Hold one end of a nichrome wire in the flame. Note its appearance while hot. Let it cool. Compare the heated end with the unheated part. What kind of change is caused by heating in this instance? Give evidence.

b. By means of tongs or pincers introduce the end of a piece of magnesium wire into the Bunsen flame. What kind of change is observed?

c. Add 2 g. of iron filings to 10 ml. of dilute hydrochloric acid in a small beaker. Note all actions. Warm the acid, if necessary, to hasten the reaction. Allow the action to proceed for ten minutes. If all the filings are used up, add more until an excess is present and the reaction is at an end. Filter the mixture into a test tube. Pour the clear filtrate into a clean evaporating dish and evaporate it barely to dryness, using a low flame. What kind of change takes place? Give evidence.

d. Obtain small strips of the following metals: iron, copper, zinc, lead. Note the color of a freshly scraped portion of each. Why does it not retain this color? Try to melt the metals in the flame of the Bunsen burner. Account for any changes in color. What metals do not tarnish in the air?

Melt a little granulated zinc in a test tube and pour it from a height on a cold iron surface. Try to burn the resulting thin metal. Note all the changes that occur. Does lead behave similarly?

Try the hardness of each specimen and arrange in the order of hardness. Try the solubility of each of these metals in water and in hydrochloric acid.

In making solubility tests, place a fragment of the substance to be examined in a test tube containing 1 ml. of cold distilled water. Shake the contents and see if anything dissolves. If cold water is without effect, heat to boiling. In like manner, try the action of hydrochloric acid on these metals, using first diluted (one part concentrated acid to five parts water), then concentrated, acid. Tabulate the results of your experiments as follows:

METAL	COLOR	MELTING POINT	HARDNESS	SOLUBILITY IN WATER	SOLUBILITY IN ACID

Why are these properties known as "specific properties"? What use do we make of these properties? How could you separate copper from a mixture of filings of zinc and copper?

All these specimens possess weight, form, and volume. Why are these properties, or attributes, not characteristic? Is temperature a specific property? May the temperature of a piece of iron be changed without altering its substance? Is this true of any specific property of iron?

e. Grind 1 g. of sugar in a mortar. What kind of change occurs? Place the sugar in a clean dry test tube and heat it slowly in the Bunsen flame. Describe the changes that occur. Test the combustibility of any gas given off. Continue heating till there is no further change. What is the residue? Is sugar to be classed as an elementary or compound substance? Why?

Grind 0.5 g. of limestone in your mortar. Take a portion of this ground material on a watch glass, moisten it with a few drops of water, and test it with a piece of red litmus paper. Give result. Strongly heat the other portion in a crucible *A* supported on a clay triangle *B* (Fig. 18). Cool the crucible, treat the contents with water, and test the aqueous solution with litmus. Give results and explain.

Seal up the end of a piece of narrow tubing about six inches long, thus making a small test tube out of it. Introduce sodium hydrogen carbonate into the tube to the depth of about one-half inch. Bend the tube at right angles about two inches from the closed end. Allow the open end to dip into a few milliliters of limewater contained in another test tube. Heat the closed end gently. Observe carefully what happens. When there is no further change, shake the ignited mass out of the tube. Receive it on a watch glass, moisten with water, and test it with red litmus. What is the product? What important industry is based on this reaction?

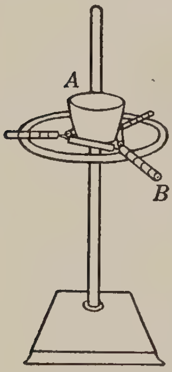

FIG. 18

15. Elements, Mixtures, and Compounds. *a.* Obtain 1 g. of sulfur from the side shelf. Record its specific properties. Obtain some copper wool. Record its properties. Place the sulfur in a test tube. Introduce into the tube a ball of copper wool of such size that it fits the tube snugly and remains half an inch above the sulfur. Heat the sulfur until it boils and its vapor comes in contact with the wool. What happens? Note the glow when the sulfur reaches the copper. This is evidence that heat energy is being given out. Examine the product. Is it more or less brittle than copper? Is it the same color as the copper? as the sulfur? The copper and sulfur have combined chemically to form a compound, copper sulfide (CuS). The equation is,

$$Cu + S \longrightarrow CuS$$

b. Place a small drop of mercury in a porcelain mortar and add to it an equal bulk of powdered iodine. Rub the mixture with a pestle. Note the color of the product. If a bright color is not obtained, allow the material to stand till the next period. The substance is mercuric iodide (HgI_2). From what elements was it formed?

$$\begin{array}{ccccc} \text{Mercury} & + & \text{iodine} & \longrightarrow & \text{mercuric iodide} \\ Hg & + & I_2 & \longrightarrow & HgI_2 \end{array}$$

What is a compound? What is an element?

19
20

c. Obtain small amounts of sulfur and of salt (NaCl) and mix them together on a watch glass. Transfer some of this material to a test tube and treat with water. Shake and allow to settle. Filter off the clear liquid into an evaporating dish. Place it on a ring supported on a ring stand and heat it carefully until the water has all evaporated. Note the color of the residue. Taste it. What is it? What specific property of the salt was involved in this separation? What is a mixture?

16. Conservation of Mass. Obtain a 4-inch test tube which may be placed within a 250-ml. Erlenmeyer flask (Fig. 19). Fit the flask with a solid stopper. Place in the flask 10 ml. of a lead nitrate solution and in the test tube 5 ml. of a solution of potassium iodide. Assemble the apparatus and stopper the flask. Be careful that the test tube is supported in an upright position. Then weigh the apparatus and record its weight. Tip the flask so that the liquid from the test tube is mixed with that in the flask. Account for the observed change. Again weigh the apparatus. Has the weight changed? Explain. What is the law of conservation of mass? How is it illustrated by this experiment?

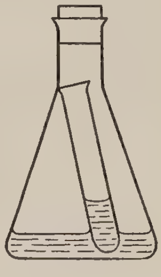

FIG. 19

17. Law of Constant Composition. Weigh accurately a 50-ml. flask. Place in it about 0.2 g. of metallic copper and again weigh accurately. Record the exact weights. Add carefully 10 ml. of dilute nitric acid (*HOOD!*). If the action does not commence add three drops of concentrated nitric acid. Cover the flask with a watch glass and allow the action to proceed. As the action slackens, add, from time to time, a few drops (about 2 ml. in all) of concentrated nitric acid. Continue these additions until the copper has completely disappeared. Do not remove the watch glass but evaporate the acid carefully until it is all gone. *Use a low flame.* When the material in the flask is free from water, gradually increase the heat until the material starts to decompose. The decomposition is indicated by the evolution of red fumes.

When no more red fumes are evolved, allow the flask to cool and then wash any material on the watch glass into the flask.

Use distilled water from the wash bottle. Evaporate carefully as before and then heat the flask almost to dull redness. Allow the flask to cool and weigh it accurately. What caused the increase in weight? Explain. Repeat the experiment, using a different weight of copper but not more than 0.3 g. and 15 ml. of acid.

RECORD

	No. 1	No. 2
Weight of flask	------	------
Weight of flask and copper . . .	------	------
Weight of copper	------	------
Weight of flask and oxide . . .	------	------
Weight of oxide	------	------
Weight of oxygen	------	------
Per cent of copper	------	------
Per cent of oxygen	------	------

What is the law of constant composition?

EXERCISES

1. How do we distinguish a chemical change from a physical change?

2. Make a list of chemical changes observed in everyday life.

3. What is the meaning of calcination? Is the conversion of limestone to lime calcination?

4. Devise an experiment to illustrate the law of conservation of mass.

5. Explain how your results in Experiment 17 illustrate the law of constant composition.

CHAPTER II

OXYGEN

NOTE. Beginning with the experiments on oxygen, your notes should include equations for all the chemical changes that you observe.

18. Sources of Oxygen. *a.* Place 0.5 g. of mercuric oxide in the bottom of a clean test tube. Clamp the tube in a test-tube holder and heat it in the flame of a Bunsen burner. While heating the oxide, insert a glowing splint into the tube from time to time, *close* to the material but *not in contact with it,* and note what happens. Notice whether any changes in the oxide take place during the heating.

b. Repeat, using 0.5 g. of lead dioxide in place of mercuric oxide. Do you observe any action?

c. Repeat *b,* using in place of lead dioxide 0.5 g. each of cupric oxide, lead oxide (litharge), iron oxide, and calcium or barium oxide. Separate test tubes are to be used for each compound. Do all simple oxides give off oxygen when heated? Take care that the splint does not come in actual contact with the substance being heated.

d. Repeat *c,* using in place of the oxides 1 g. each of the following in separate test tubes: potassium chlorate, potassium nitrate, and manganese dioxide. When manganese dioxide is tested, a hard-glass test tube and the flame from either a Tirrill burner or a blast lamp should be used. What general conclusions do you draw from *d*?

e. Place 0.5 g. of sodium peroxide in a test tube, add about 5 ml. of water, and test the evolved gas with a glowing splint. What result is observed?

f. Which substance in *a, b, c, d, e,* is best suited for preparing oxygen in quantity?

19. Catalysis. *a.* Place 1 g. of potassium chlorate in a test tube. Clamp the tube in a vertical position to a ring stand and heat the chlorate gently until it *just* melts. Test the escaping gas by means of a glowing splinter. Is any oxygen given off? By means of a knife blade or a spatula, drop a minute quantity of manganese dioxide into the molten substance. (Keep your eyes away from the mouth of the tube.) Test the escaping gas with a glowing splinter. What is the effect of the dioxide? What name is applied to substances which influence chemical reactions in this way?

b. Try the solubility of potassium chloride, potassium chlorate, and manganese dioxide, respectively, in water. How could you show that manganese dioxide remains unchanged after the action in *a*, and that the oxygen comes entirely from the potassium chlorate?

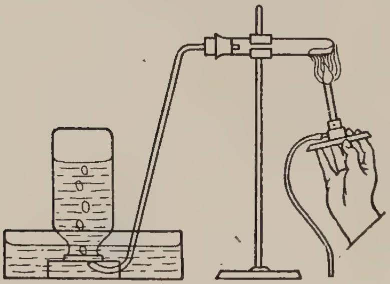

FIG. 20

20. Laboratory Preparation of Oxygen. Calculate the amount of potassium chlorate necessary to liberate a liter of oxygen under standard conditions. Mix the calculated amount of potassium chlorate with one fourth its weight of manganese dioxide. If a small portion of this mixture, heated rapidly in a test tube, decomposes quietly, the remainder can be safely used in the preparation of oxygen.

Place the remainder of the mixture in a test tube provided with a one-hole cork or rubber stopper and a delivery tube. Clamp the tube in a nearly horizontal position on a ring stand and connect it to a pneumatic trough by means of a glass delivery tube (Fig. 20). Fill four wide-mouthed bottles with water, cover the tops with glass plates, and invert them in a trough containing water. Gently heat the tube containing the mixture, starting from the lower end of the tube. Hold the burner in the hand and heat evenly all parts of the tube containing the mixture. When the bubbles begin to escape and all the air is out of the apparatus, the end of the delivery tube is brought under one of the inverted bottles. The oxygen rises in the bottle and displaces the water. When the water has all been displaced, slip a glass plate under the mouth of the bottle and hold it there firmly with the hand. Remove the bottle and place it upright on the desk. Why?

When one bottle is filled, place the delivery tube under the second bottle of water. Remove the delivery tube from the water whenever you stop heating, or when the bubbles cease coming off. Why?

Note the physical properties of oxygen. What do you observe as to physical state, color, odor, solubility in water? Use the bottles of oxygen in the following experiments.

21. Chemical Activity of Oxygen. *a.* Burn sulfur in a deflagrating spoon and give the odor of the product. Note the color and size of the flame. Lower the burning sulfur into a bottle of oxygen. Is there any difference in the way in which sulfur burns in air and in oxygen? Is the odor the same? What conclusion do you draw from this fact? Add 10 ml. of distilled water to the bottle in which the sulfur was burned, cover the bottle, and shake it well. Test the solution with blue litmus paper. Test distilled water in the same way. Give the result in each case.

b. Remove all sulfur from the spoon and then repeat *a,* using a very small piece of *red* phosphorus in place of the sulfur. Why does the product form a smoke, instead of remaining in the spoon? Make a solution of the product in water and test the liquid with litmus.

c. Place a piece of charcoal in the spoon, hold it in the flame until it glows, and lower the glowing charcoal into a bottle of oxygen. Cover the mouth of the bottle with a glass plate. Add *clear* limewater and shake the bottle. What happens?

d. In a clean deflagrating spoon place an exceedingly small piece of sodium (*storeroom*). Ignite it and insert the spoon into a bottle of oxygen. After the sodium has burned, add 10 ml. of water to the bottle. Dip the spoon in the water. Test the solution with *red* litmus paper. What result is observed?

22. Rate of Oxidation. Moisten 1 g. of iron filings or powdered iron and place them in the bottom of a test tube containing 2 ml. of water. Invert the test tube in a beaker of water and mark the level of the water in the test tube with a rubber band. Allow it to stand until the next laboratory period. Note then what has happened. Test the water with red and with blue litmus paper.

Hold an eightpenny nail in the flame of the Bunsen burner until it is red-hot. Remove and note the scale on the outside of the nail. What is this scale? How was it formed? Hold a bundle of small iron wires (from 30 to 40 gauge) in the flame in a similar way. Explain the observed changes. Why is the action different from that with the nail? Sprinkle into the Bunsen flame a little powdered iron. Describe the observed phenomena. What factors are here shown to affect the rate of oxidation? What other factors will also affect the rate of oxidation?

EXERCISES

1. From your experiments do you reach the conclusion that a substance has a definite kindling temperature? Give evidence for your answer.

2. Which of the chemical changes observed are combination? Which are decomposition?

3. Account for the fact that a substance burns more vigorously in pure oxygen than in air.

4. What weight of oxygen is required to burn 5 g. of sulfur to sulfur dioxide?

5. Do we increase the affinity of wood for oxygen by cutting a piece of it into fine shavings?

6. Why is dust in mills or mines often a source of danger?

CHAPTER III

HYDROGEN

23. Preparation and Detection of Hydrogen. (*Optional. To be performed only under the direction of an instructor.*) *a.* Fill a pneumatic trough (a stone crock makes a good substitute) nearly full of water and invert in it a test tube full of water. Obtain from the storeroom a small piece of sodium and note its physical properties. Wrap about it a piece of filter paper wet with kerosene and so arranged that only three sides of the sodium are covered. Grip this with your crucible tongs and place it under the mouth of the inverted test tube (Fig. 21). The sodium can thus be introduced into the test tube. It will react with the water, liberating hydrogen. The gas will displace the water and fill the tube. When the tube is full of hydrogen, bring its mouth to a flame. What result is observed? This burning, sometimes with explosive violence, is the usual test for hydrogen. Note the soapy-like feeling of the liquid and test its effect on blue litmus and on red. Sodium hydroxide is formed by the action of sodium on water:

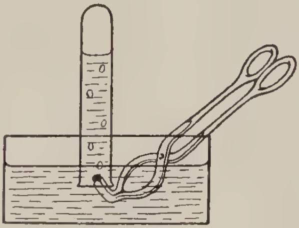

FIG. 21

sodium + water ⟶ sodium hydroxide + hydrogen

b. Place a 4-inch ribbon of magnesium in a test tube and pour on it 2 ml. of a dilute solution of sulfuric acid. Note the chemical action. Test the escaping gas with a *lighted* splint. What is the gas? What type of chemical change is illustrated? Explain.

c. Test in a similar manner, in separate tubes, single pieces of zinc, aluminum, iron, and copper. Which of these liberate hydrogen from sulfuric acid? Give the equations.

d. Place a small amount of aluminum in a test tube and add dilute sodium hydroxide. Test the escaping gas for hydrogen. Apply heat, if necessary. Give the equation.

24. Laboratory Preparation of Hydrogen. Fit a bottle *A* (Fig. 22) with a two-hole rubber stopper bearing a thistle tube *B* and a glass tube *C* about 15 cm. long and bent at right angles. Support the bottle on the base of a ring stand and secure it in position with a clamp. Now invert a wide-mouthed bottle full of water on the shelf of the pneumatic trough (this can be done best by filling the bottle with water, placing over its mouth a glass plate, inverting the bottle over the shelf, and removing the plate). Connect with the right-angled tube a delivery tube *D*, using a short piece of rubber tubing.

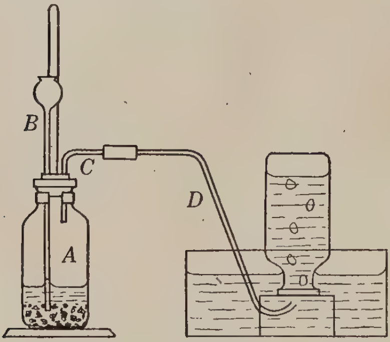

FIG. 22

NOTE. The delivery tube has an obtuse bend near the bottle and an acute bend near the other end, which enable the gas to bubble through the hole in the shelf of the trough. These tubes must be nicely made, with smooth, well-rounded bends and fire-polished ends. The glass tubes must touch each other within the rubber tubing. Poorly made or poorly erected pieces will be dismantled by the laboratory instructors.

Place from 10 to 15 g. of granular zinc in the bottle, stopper it tightly, adjust the thistle tube till it comes nearly to the bottom of the bottle (why?), and pour dilute sulfuric acid through the thistle tube. When action starts, test the escaping gas by displacing the water from an inverted test tube and lighting the gas thus collected. Repeat. A *distinct flame* must be seen, and there must be no *explosion* before the gas is collected in the bottles. Since a mixture of hydrogen and air is highly explosive, all flames must be kept away from the generator. Wrap the bottle in a towel. When the gas burns quietly, collect a bottle full. What is the color of hydrogen? its odor? its taste? Have ready a *clean dry* watch glass. Now light the hydrogen in one of the bottles. Hold the watch glass just above the mouth of the bottle (do not shut off the air). What causes the cloud which appears on the glass? Where does this substance come from?

25. Hydrogen Flame; Synthesis of Water. Remove the delivery tube and put in its place a calcium chloride tube (Fig. 23) about half filled with granular calcium chloride (this frees the hydrogen from moisture). Place in the large end of the drying tube a one-hole stopper fitted with a glass tube bent at right angles and with the outermost end drawn to a jet. Make sure that the apparatus is air-tight and test the gas coming from the jet by collecting it in a test tube as directed in the previous experiment. When the hydrogen collected in the test tube burns quietly and smoothly, transfer the flame by means of a test tube to the jet. Give the color of the flame as it appears at first, and after a few seconds. Explain. Hold a *clean dry* beaker near the flame. Note the film of moisture. Where does this water come from? Test the temperature of the flame by holding in it a piece of iron wire. What industrial use is made of the oxyhydrogen flame?

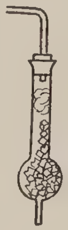

FIG. 23

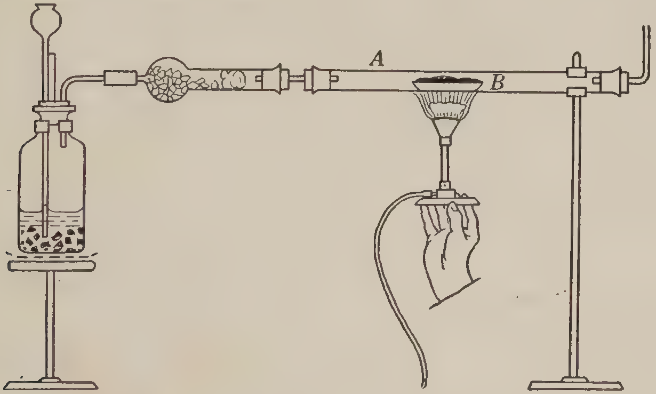

FIG. 24

26. Reduction by Hydrogen. Remove the glass jet and, by means of a one-hole stopper fitted with a glass tube (Fig. 24), connect the calcium chloride tube with a piece of hard-glass combustion tubing *A* about 25 cm. long. Introduce into the combustion tubing a porcelain boat *B* about half full of copper oxide (previously heated). Note the physical properties of the copper oxide. Place in the opposite end of the combustion tube a one-hole stopper bearing the glass jet. Tighten up the stoppers and test the hydrogen escaping from the jet by collecting it in a test tube as in Experiment 24. When all air is expelled, as evidenced by a quiet flame, heat the tubing near the boat for several minutes with a Bunsen burner. Note the moisture condensing on the cooler portions of the tube. What is its source? Does the material in the boat change in color? Why? Let the tube cool down and then examine the residue in the boat. What name is given to this type of chemical action?

27. Activity of Metals. Test separately the action of cold water, boiling water, and dilute hydrochloric acid on calcium, magnesium, zinc, iron, and copper. Tabulate your results according to the following scheme:

	COLD WATER	HOT WATER	ACID
Calcium	------------	------------	------------
Magnesium	------------	------------	------------
Zinc	------------	------------	------------
Iron	------------	------------	------------
Copper	------------	------------	------------

EXERCISES

1. What stops the acid from acting on zinc when the delivery tube of a Kipp generator is closed?

2. Name three metals which do not liberate hydrogen from acids.

3. What is displacement?

4. Why does hydrogen not ignite when allowed to escape into the air?

5. Should you expect hydrogen to reduce calcium oxide to metallic calcium?

CHAPTER IV

ATOMIC THEORY, SYMBOLS, FORMULAS, AND EQUATIONS

28. Derivation of the Formula of Cuprous Oxide. Set up an apparatus like the one used in Experiment 26 for the reduction of copper oxide. Weigh a porcelain boat accurately. Place in it about 0.5 g. of cuprous oxide. Spread it out along the boat. Weigh the boat and contents. Insert the boat carefully in the combustion tube. Using the same precautions as in Experiment 26 (what are they?), heat the boat and contents while passing over it a constant stream of pure hydrogen. When the action seems complete, allow the tube to cool. Then remove the boat and contents and weigh accurately. Place the boat back in the tube and repeat the heating (more zinc may be needed in the generator). Cool the tube and reweigh the boat. If the weight remains the same, no further reduction is possible; if a decrease in weight is noted, the heating in hydrogen must be continued until a constant weight is obtained. Tabulate all weighings. What caused the decrease in weight? What substance is left in the tube? Calculate the per cent of copper and of oxygen in the original substance.

RECORD

Weight of boat ------------------------------
Weight of boat and cuprous oxide ----------------
Weight of cuprous oxide ----------------------
Weight of boat and copper
    First weight ----------------------------
    Second weight ---------------------------
Weight of copper ---------------------------
Weight of oxygen ---------------------------
Per cent of copper --------------------------
Per cent of oxygen --------------------------
Formula of cuprous oxide ---------------------

3200   3800

EXERCISES

1. What is the law of combining weights?

2. Explain how the atomic theory gives a logical explanation of the law of combining weights.

3. Who originated the practice of using symbols to express the atomic weights of elements?

4. What information is given by the formula derived for the oxide?

5. A compound is known to contain 82.35 per cent of nitrogen and 17.65 per cent of hydrogen. Calculate the simplest possible formula.

6. A compound has the following composition: carbon, 76.58 per cent; hydrogen, 6.40 per cent; oxygen, 17.02 per cent. Calculate the simplest formula.

7. A substance is found to contain 38.55 per cent of oxygen, 46.97 per cent of potassium, and 14.48 per cent of carbon. What is its simplest formula?

8. Calculate the per cent of each element present in each of the following compounds:

Potassium chromate, K_2CrO_4
Lead nitrate, $Pb(NO_3)_2$
Sulfuric acid, H_2SO_4

CHAPTER V

WATER AND HYDROGEN PEROXIDE

29. Distillation of Water. Fit a 750-milliliter distilling flask *A* with a rubber stopper bearing a thermometer *B*. Place it on a wire gauze supported on a ring and clamp it in place (Fig. 25). Fill it half full of water. By means of a one-hole stopper connect the delivery tube with a Liebig condenser *C* supported by a ring stand. Attach the condenser to the water tap by a rubber tube and circulate cold water through the outer part of the condenser. The water should enter the lower opening and flow out of the upper one. Why? Place a flask under the lower end of the condenser. Adjust the thermometer as shown in the figure. Apply heat to the flask. What is the temperature when water first starts dropping into the flask? Collect about 50 ml. of "distilled" water. Taste it. Has the taste changed?

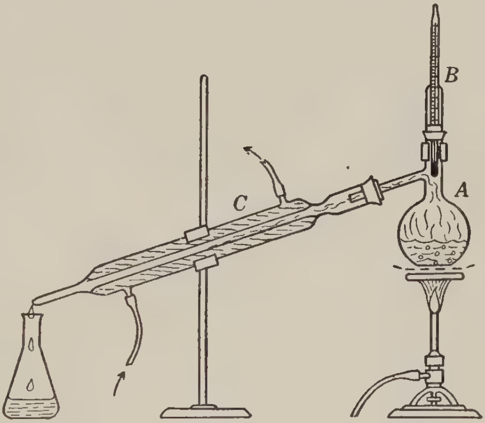

FIG. 25

30. Purity of Distilled Water. Pour some of the distilled water into a clean watch glass and place it over a beaker nearly full of water. Apply heat to the beaker and evaporate the distilled water on this "water bath." Note whether any residue is left on the watch glass. Repeat, using tap water. Note the residue. Why is there this difference between distilled water and tap water? What impurities would leave no trace of their presence in such a test? What is the purest natural water?

31. Purification of Water. Fill a small beaker about half full of water and add enough litmus solution to give a distinct blue color. Divide the water into three portions. To one add powdered charcoal, heat the water to boiling, and filter. Note the color of the filtrate. To the second portion add about 5 ml. of aluminum sulfate (or alum) solution and an equal volume of dilute ammonium hydroxide. Note the white precipitate. Does the color of the precipitate change as it settles? Does the color of the solution change? Filter, and note the color of the filtrate.

32. Chemical Properties of Water. Test the action of litmus on distilled water. Place a small lump of calcium oxide in a test tube and add distilled water. Shake the mixture and filter off any residue. Test the filtrate with litmus. What kind of an oxide is calcium oxide? Write the equation for the action of water on the oxide.

Blow your breath through a glass tube into a clear solution of calcium hydroxide. What result is observed? Give the equation. What kind of an oxide is carbon dioxide?

Test the action of metallic calcium on water. Give the equation.

33. Water of Hydration. Heat carefully, in separate test tubes, a few small *crystals* of each of the following substances: sodium sulfate, borax, and copper sulfate. What are the results? Explain the deposition of water on the cooler portion of each test tube. In a similar way heat a few crystals of potassium dichromate. Do all crystalline substances contain water of hydration? Now add hot water to the tube containing dehydrated cupric sulfate. What is the result?

34. Determination of the Percentage of Water of Hydration. Weigh accurately a clean dry porcelain crucible and cover. Place in it about 2 or 3 g. of crystals of barium chloride, and weigh. Place the covered crucible on a pipestem triangle supported on a ring stand and heat carefully with a very low flame for five minutes. Then heat the crucible strongly for about twenty minutes. Allow it to cool, and weigh. Heat the crucible and contents for another five-minute period. Allow it to cool, and weigh it as before. If a loss of weight is noted, reheat and reweigh until a constant weight is obtained. Record all weighings as follows:

RECORD

Weight of crucible --------------------------------
Weight of crucible and chloride ---------------------
Weight of hydrated chloride ------------------------
Weight of crucible and contents after heating
  First weight -----------------------------------
  Second weight ----------------------------------
Weight of the anhydrous chloride -------------------
Weight of water driven off -------------------------
Per cent of water in the hydrate --------------------
Formula of the hydrate -----------------------------

35. Hydrogen Peroxide. *a.* Sift about **1** g. of sodium peroxide into a flask containing 100 ml. of water. Cool the flask under running water while adding slowly dilute sulfuric acid until a drop of the solution turns blue litmus paper red. The solution thus prepared contains hydrogen peroxide.

b. Make some starch iodide paper by soaking strips of filter paper first in potassium iodide solution and then in starch paste. Place on one of these papers a few drops of the solution from *a*, acidified with sulfuric acid. A deep blue color results. This is one test for hydrogen peroxide.

c. Dilute 1 ml. of lead nitrate solution to 10 ml. and saturate it with hydrogen sulfide, using a precipitation flask (Fig. 26). Remove the stopper once or twice to allow air to escape, otherwise keep the flask tightly closed. This is absolutely essential, since hydrogen sulfide is poisonous. Filter off the black precipitate and wash it once with hot water. Place the paper and precipitate in a beaker and add enough hydrogen peroxide to cover the paper. Stir the mixture. What color change is noted? How do you account for this change? Give the equation.

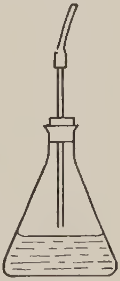

Fig. 26

d. Place about 1 ml. of potassium dichromate solution in a test tube and make the liquid slightly acid with dilute sulfuric acid. In a second test tube place 5 ml. of hydrogen peroxide and add 3 ml. of ether. Shake the tube and allow the ether to separate. Then add a few drops of the acidulated dichromate solution and shake again. As the upper layer separates note its blue color. This is used as a test for hydrogen peroxide and for chromates and dichromates.

e. Place in a test tube 3 ml. of hydrogen peroxide. Add 0.5 g. of powdered manganese dioxide. Test the gas evolved with a glowing splint. What is the result? Explain. Repeat this experiment, using some finely powdered metal. Do you note any difference? Test the effect of finely powdered charcoal. Explain your results.

EXERCISES

1. Name several oxides which give acids on treatment with water; several which give bases.

2. How many calories of heat are required to melt 15 g. of ice at 0° without changing its temperature? How much heat is used up in converting 17 g. of water at its boiling-point into steam at the same temperature?

3. How much water, in grams, is required to hydrate 10 g. of $CuSO_4$ completely? (The hydrated salt is $CuSO_4 \cdot 5\ H_2O$.)

4. How much oxygen may be obtained from the action of 10 g. of 4-per-cent hydrogen peroxide on silver oxide? How much oxygen would the same peroxide yield when completely decomposed by catalysis?

5. What should you obtain by leading sulfur dioxide into hydrogen peroxide?

6. Show how the two oxides of copper illustrate the law of multiple proportions.

CHAPTER VI

EQUIVALENT WEIGHTS AND VALENCE

36. Equivalent Weight of Iron in Ferric Oxide. Ferric oxide is made by dissolving iron in nitric acid and decomposing the ferric nitrate formed during the reaction.

Weigh accurately about half a gram of pure iron wire well cleaned with sandpaper. Introduce the iron into a 50-milliliter Erlenmeyer flask (pyrex) and weigh both together. Add 10 ml. of dilute nitric acid and allow the iron to dissolve completely. Evaporate the solution to dryness, taking special care to avoid spattering. When the product is dry, heat the flask more strongly until no more red fumes come off and all the nitrate is changed into the oxide. Cool the flask and then obtain its weight. Tabulate your results as follows:

RECORD

Weight of iron, a ----------------------------------
Weight of flask and iron, b -------------------------
Weight of flask, $b - a$ -----------------------------
Weight of flask and oxide, c ------------------------
Weight of oxide, $c - (b - a)$ ------------------------
Weight of oxygen, $c - b$ -----------------------------
Equivalent weight of iron ---------------------------
Valence of iron ------------------------------------

EXERCISES

1. Do some elements have more than one equivalent weight?

2. Calculate the percentage composition of ferric oxide, using the data obtained by experiment.

3. From the percentage composition determine the simplest formula of the oxide.

4. What is the valence of oxygen?

CHAPTER VII

THE ATMOSPHERE

37. Determination of the Relative Volumes of the Active and Inactive Components of the Atmosphere. Place about 0.2 g. of white phosphorus in a slender dry test tube (use forceps, not the fingers, in handling phosphorus). Cover the mouth of the tube with a wet filter paper held firmly in place with the thumb. Warm the tube until the phosphorus ignites. Allow the burning to continue until the oxygen is used up. Being careful not to let the hot phosphorus come in contact with the thumb, immerse the test tube in water in a large beaker, remove the thumb and paper, and quickly invert the tube. After allowing time for the tube to cool, depress it until the water level is the same without and within (why?). Mark the tube at the water level, remove it, and fill it up to the mark with water. Measure with a graduate the volume of water used. Measure the volume of water needed to finish filling the tube. From these values calculate the per cent by volume of oxygen in air. Tabulate your results.

38. Carbon Dioxide in Air. By using an apparatus of your own design, draw air through limewater. What is the precipitate? Give the equation.

39. Water in Air. Allow a stick of sodium hydroxide half an inch in length to remain exposed to the air in the laboratory. After an hour observe what has taken place, and explain.

40. Determination of the Dew Point of Air. Fill a polished metal cup about one third full of distilled water. Bring the water to the temperature of the laboratory by warming or cooling it as the case demands. Read accurately its temperature. Add a few pieces of ice and stir the mixture carefully with the thermometer. Watch for the formation of a mist on the outer surface of the cup (do not breathe on the cup). Further cool the water, if necessary, by adding more ice until the mist starts to form. Observe the temperature. Warm the water in the cup by adding small amounts of warm water. Stir the water carefully. Observe the temperature when the mist disappears. Repeat the experiment until values are obtained which do not vary by more than two or three degrees. The average of these values represents the dew point of the air. Calculate the relative humidity of the air in the laboratory.

EXERCISES

1. What useful functions have the dust particles in the air?

2. Which of the components of the atmosphere should you expect to find increased in the air over large industrial centers? Why?

3. Discuss the carbon dioxide and oxygen cycle in nature. Illustrate the idea from your own experience.

4. Who first explained the correct rôle of oxygen in respiration?

5. What is the composition of air by weight?

6. What should you expect to be the result if plants were grown in an atmosphere containing 20 per cent of carbon dioxide?

7. Why are we likely to suffer from too high a humidity in summer, whereas the converse of this is true in winter?

8. Why is a room with a low humidity uncomfortable at 68° F.? Why is it made comfortable by raising the humidity?

CHAPTER VIII

THE GAS LAWS

41. The Gas Laws and Pressure and Volume. Clean a 100-milliliter graduated cylinder and fill it one third full of water. Place a wet filter paper over the end of the graduate and invert it in a tall beaker three fourths filled with water (Fig. 27). Fasten it in such a position that the level of the water in the graduate is above that in the beaker. Read the volume of the inclosed air. Now release the clamp and adjust the graduate so that the level of the water inside is below the level of the water in the outer vessel. Read the volume of the inclosed air as before. Why is there a change in volume? What are the conditions chosen as standard in the measurements of gases? Now adjust the graduate so that you can read the volume of the inclosed air at the pressure of the atmosphere in the room. How do you know when such a position is reached? Is the air in the graduate perfectly dry? Explain your answer. Calculate the volume the air would occupy when dry at the temperature and pressure of the laboratory. Calculate the volume of air under standard conditions.

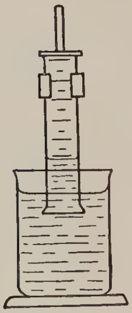
FIG. 27

42. Charles's Law. When the pressure is constant, the volume of a gas varies directly as the absolute temperature. In the following experiment a given volume of air measured at the boiling-point of water is allowed to cool to room temperature. The volume at the room temperature will evidently be less than at the boiling-point of water. The diminution in volume may be measured by allowing water to enter the containing vessel until the pressures are equalized.

Select a clean dry flask *A* having a capacity of approximately 250 ml. Fit it with a one-hole rubber stopper bearing a glass tube *B* about 8 cm. long (Fig. 28). The tube should have its ends fire-polished. Fit it with a short piece of rubber tubing carrying a pinch clamp *C*. Immerse the flask in water to the top of its neck and open the pinch clamp. Boil the water in the beaker *D* for about ten minutes. Record the temperature of the boiling water (do not touch the side of the beaker with the thermometer bulb when making this temperature reading). Close the pinch clamp tightly and remove the flask. Invert the flask over water in a pneumatic trough.

Release the pinch clamp and allow water to run into the flask. Keep the flask in the trough until the temperatures are equalized (about ten minutes); then adjust the flask so that the water levels inside and outside are the same, and close the pinch clamp. Remove the flask from the trough, place it in an upright position on the desk, and carefully mark the position of the stopper in its neck. Remove the stopper, and measure with a graduate the amount of water which entered the flask. Then find the amount of water required to fill the flask and the tube when the stopper is at the indicated point. The total volume of the flask represents the volume of air at the boiling-point of water. The total volume of the flask, minus the volume of water entering it on cooling, gives the volume of air plus water vapor, at the temperature of the water in the pneumatic trough. Look up the aqueous tension at the temperature of the water, and calculate the volume of dry air at laboratory pressure. Compare this value with the volume that the air should occupy according to Charles's law. Calculate your per cent of error.

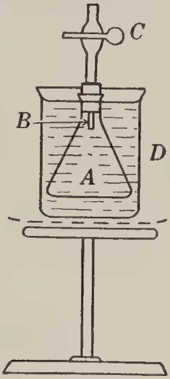

FIG. 28

In carrying out this experiment it is essential to allow the flask to remain a sufficient length of time in both the hot and the cold water to equalize the temperatures. If any air is allowed to enter the flask during the process of removing it from the water bath, your per cent of error will be very high.

43. Diffusion. *a.* Fill a bottle with hydrogen and another with oxygen. Bring their mouths together as shown in Fig. 29. Allow them to remain in this position for two minutes and apply a burning splint to the mouth of each. Give the results and explain. What is the relative rate of diffusion of the two gases?

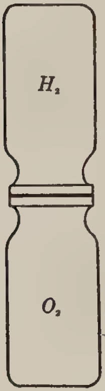

FIG. 29

b. Remove the stoppers from bottles of concentrated hydrochloric acid and ammonium hydroxide. Stand the bottles six inches apart and wave your hand back and forth above them. Note what happens. Stopper the bottles for a time and then open them, but do not disturb the air over them. Does the result obtained differ in any respect from the previous one? Explain.

Moisten a stopper with the concentrated acid and another with the concentrated hydroxide. Quickly bring these stoppers within an inch of each other. Note where the fumes arise. Repeat several times to be sure that your first observation was a correct one. Explain your observations. State which has the greater density: hydrogen chloride (HCl) or ammonia (NH_3).

EXERCISES

1. Do the gas laws hold rigidly for all gases?

2. If a gas occupies a volume of 100 cu. ft. at 10°, what volume will it occupy at 293°, provided the pressure remains constant?

3. To what temperature must a gas be heated in order that its volume at − 53° may be doubled?

4. A gas occupies a volume of 100 cu. in. under a pressure of 15 lb. per square inch. What pressure must be placed on it to decrease its volume to 75 cu. in.?

5. A gas occupies a volume of 1000 ml. What volume will it occupy if the pressure be increased four times?

6. If 2000 ml. of a gas is collected at − 20° and a pressure of 720 mm., what volume will it occupy at 233° and a pressure of 1440 mm.?

7. What volume will 100 ml. of a gas measured at 30° and 760 mm. collected over water occupy in the dry condition measured at 20° and 780 mm.?

8. Oxygen is sold in containers under a pressure of 1800 lb. at 21°. What will be the pressure when the temperature increases to 30°?

9. What volume will 100 ml. of a dry gas measured at 380 mm. and 10° occupy when collected over water at 20° and 760 mm.?

10. 10 ml. of a gas measured at 380 mm. and 273° weighs 0.1 g. What will be the weight of 10 ml. of the gas measured at 760 mm. and 0°?

11. 500 ml. of oxygen is collected at 273° and a pressure of 380 mm. What volume will the gas fill at standard conditions?

12. A steel cylinder having a volume of 600 cu. ft. is filled with a gas at 20° and a pressure of one atmosphere. The temperature is raised to 313°. If the volume remains unchanged, what will the pressure be?

CHAPTER IX

MOLECULAR WEIGHTS AND ATOMIC WEIGHTS

44. Determination of the Molecular Weight of Oxygen. Set up the apparatus shown in Fig. 30, consisting of a hard-glass test tube *A* (pyrex), a 750-milliliter Florence flask *B* nearly filled with water, and a rubber connection *C* bearing a pinch clamp *D* and ending in a glass tube *E* drawn out to a jet. Be sure that all glass bends are well rounded and that all connections are tight. Rubber stoppers must be used.

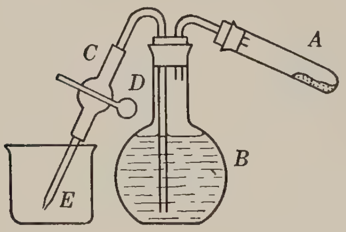

FIG. 30

Place in the test tube *about* 3 g. of well-powdered potassium chlorate ($KClO_3$). Add about 0.2 g. of manganese dioxide (MnO_2). Mix these substances thoroughly by shaking and jarring the tube. Assemble the apparatus.

Dry the mixture by heating the test tube very gently with the flame of a Bunsen burner (*CARE!*). Have the pinchcock on the outlet tube open (why?). As moisture appears on the side of the tube, drive it upward by warming the tube to within one inch of the stopper. Enough heat should be applied so that some oxygen is evolved. From 50 to 100 ml. of water will usually be driven over. Allow the apparatus to cool (why?), and then close the pinchcock on the outlet tube. Disconnect the test tube and weigh it *accurately*. Record the weight, in ink, in the tabular form of record.

Replace the tube in its former position, open the pinchcock, keeping the end of the outlet tube under water, and raise the beaker until the surfaces of the water in the flask and in the beaker are at the same level. Now close the pinchcock, place a dry beaker under the outlet tube, and open the pinchcock. If water continues to siphon over, your apparatus is leaky and must be rebuilt. When the apparatus is gas-tight, heat the tube gradually until the evolution of oxygen has caused about 200 ml. of water to flow into the beaker. Allow the tube to *cool,* equalize the levels as before, and close the pinchcock. Measure, with your graduated cylinder, the volume of water driven over. Record the volume in ink in the tabular form. Now remove and weigh the test tube. Record its weight.

Replace the tube and repeat the experiment. Again allow the tube to cool, measure the volume of water, and weigh the tube. Record the values. Note the temperature and the barometric pressure. You now have two sets of data, from each of which you are to calculate the weight of 22.4 liters of oxygen under standard conditions. Show all work.

RECORD

First weight of tube ____________________
Second weight of tube ____________________
First weight of O_2 ____________________
Third weight of tube ____________________
Second weight of O_2 ____________________
First volume of water ____________________
Second volume of water ____________________
Temperature of water ____________________
Barometric pressure ____________________
Vapor pressure of water ____________________
First volume of O_2 at standard conditions __________
Second volume of O_2 at standard conditions __________
First molecular weight of O_2 ________________
Second molecular weight of O_2 ________________
Average molecular weight of oxygen ______________

45. Determination of the Equivalent Weight of a Metal. The equivalent weight of a metal may be found directly by determining the weight of hydrogen liberated when a known weight of some metal is allowed to act with certain acids. It is ordinarily neither convenient nor accurate to weigh the hydrogen evolved; hence its weight is calculated. It has been found that a liter of dry hydrogen measured under standard conditions weighs 0.08987 g.; consequently, if the volume of hydrogen evolved is known, as well as the temperature and pressure under which it was collected, we can calculate its weight.

Set up the apparatus as shown in the sketch (Fig. 31). Use a rubber stopper. The glass tubing leading from the drying tube *A* to the flask should lie in one plane, and the bends should be rounded. For the sake of neatness, stability, and convenience the ring stand should be placed with the short edge parallel to the edge of the desk. All bends must be of glass tubing.

When you have set up the apparatus and fully understand the method of carrying out the experiment, report to your laboratory instructor. If your preparation and apparatus are satisfactory, he will give you a sample of a metal. After cleaning and weighing the sample, place it in the drying tube on the copper spiral *B*. Fill the entire apparatus with water. Close the pinch clamp. Place the flask filled with water over the delivery tube, and then pour concentrated hydrochloric acid into the funnel. Open the pinch clamp and allow the acid to flow into the tube with the metal. Add acid, if necessary, to keep the funnel from becoming empty and trapping air in the tube. When hydrogen begins to be evolved at the metal, close the pinch clamp. More acid can be allowed to enter the system as needed by raising or lowering the funnel. When the pressure of the hydrogen has forced the level of the water nearly into the neck of the flask, begin to remove the acid by pouring water into the funnel and opening the clamp, taking care that no air is trapped and forced over into the flask. The level of the water should be about the middle of the neck by the time that the action ceases. Carefully dry the metal that remains by pressing it between sheets of filter paper. When dry, weigh it accurately and record the weight.

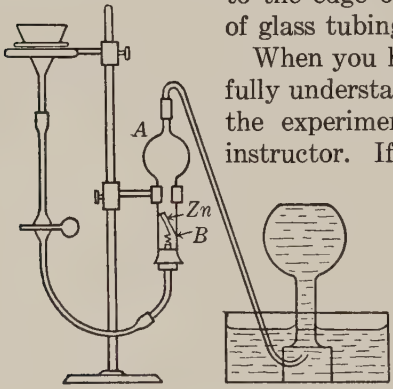

FIG. 31

Raise or lower the flask until the levels of the water on the inside and the outside are the same. To avoid heating the gas, hold the flask by the part of the neck that is under the water and make a mark on the neck corresponding to the level of the water. Determine the temperature of the hydrogen. Wash the trough thoroughly to remove the acid; then find the amount of water necessary to fill the flask to the mark. This represents the volume of hydrogen plus water vapor at laboratory conditions. Reduce this volume to that which the dry gas would occupy under standard conditions, and calculate the equivalent weight of the metal.

RECORD

First weight of metal ______________________
Second weight of metal ______________________
Weight of metal used ______________________
Volume of water ______________________
Temperature of hydrogen ______________________
Volume of hydrogen at standard conditions __________
Weight of hydrogen ______________________
Equivalent weight of the metal ______________________

46. Molecular Weight of Steam. (Adapted from the *Journal of Chemical Education*, Vol. I (1924), p. 152.) Arrange an apparatus as shown in Fig. 32. Place a 500-milliliter Erlenmeyer flask fitted with a rubber stopper in a 2-liter beaker. Fill the beaker up to the level of the stopper with a saturated brine of calcium chloride. Before heating the brine, disconnect the U-tube at *B* and *C*, and the soda lime and calcium chloride tube at *A*, the precaution being taken to close these tubes by short pieces of rubber tubing having one end closed with a plug of glass rodding. Weigh the U-tube accurately and attach it to the straight calcium chloride tube at *C*.

Open the screw clamp at *D*. Heat the brine to 106° and add 4 ml. of hot water to the flask through the tube at *D*. Allow the water to evaporate, and repeat the operation twice to expel the air from the flask completely. Continue the heating until there is no water in the flask or in the tubes.

Immediately attach the soda lime and calcium chloride tube at *A* and the U-tube at *B*, and then close the screw clamp tightly at *D*. Start the suction pump and regulate it so that only two or three bubbles of air per second pass through the drying bottle of sulfuric acid *E*. By this means dry air free from carbon dioxide is drawn through the apparatus and sweeps the steam into the U-tube. Keep the temperature of the bath constant during the operation.

After the suction has been kept up for forty minutes, disconnect the tube, close the ends with the rubber tubes carrying the glass plugs, and reweigh the tube. The increase in weight represents the weight of the steam in the flask. Measure the volume of the flask up to the lower end of the stopper. From the known weight of steam and the known volume of the flask, calculate the weight of 22.4 liters of steam under standard conditions.

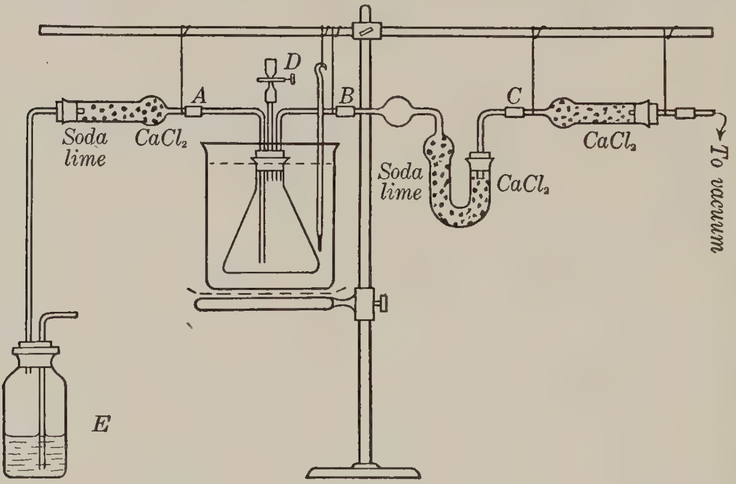

FIG. 32

RECORD

Weight of U-tube at beginning ____________________
Weight of U-tube + steam ____________________
Weight of steam ____________________
Volume of steam uncorrected ____________________
Volume of steam corrected ____________________
Weight of 22.4 liters of steam ____________________

From this value and the percentage composition of water, determine the formula of water. Compare your weight of 22.4 liters with the true molecular weight.

EXERCISES

1. Make a general statement which would indicate the metals whose equivalent weights could be determined by the method used in Experiment 45.

2. Make a general statement as to the acids which might be used in Experiment 45.

3. Give the relation between the atomic weight, the valence, and the equivalent weight of an element.

4. What is the purpose of the copper spiral used in Experiment 45?

5. 0.1008 g. of hydrogen is displaced from an acid by 11.87 g. of a certain metal. What is the equivalent weight of the metal?

6. What is the weight of 896 ml. of *dry* hydrogen measured at 273° and 380 mm. of mercury?

7. 0.5584 g. of a metal combines with 1.065 g. of chlorine. What is the equivalent weight of the metal?

8. The equivalent weight of one of the elements is 69.67, and its valence is 3. What is the atomic weight of the element? What is the formula of its hydride?

9. Write the equation for the decomposition of potassium chlorate by heat.

10. What does the equation above mean in terms of (*a*) molecules? (*b*) moles? (*c*) grams? (*d*) pounds?

11. If 30 g. of $KClO_3$ is completely decomposed by heat, calculate the volume of oxygen obtained (*a*) at standard conditions; (*b*) at 24° and 730 mm.

CHAPTER X

CHLORINE AND HYDROGEN CHLORIDE

47. Preparation of Chlorine (*HOOD!*). *a.* Place in separate test tubes very small amounts of manganese dioxide (MnO_2), potassium permanganate ($KMnO_4$), and potassium dichromate ($K_2Cr_2O_7$). Add 2 ml. of concentrated hydrochloric acid to each. Warm the tubes, if necessary, to start the action. Note the color and odor of the evolved gas. Hold in it a strip of potassium iodide starch paper. What change do you see? Write all equations.

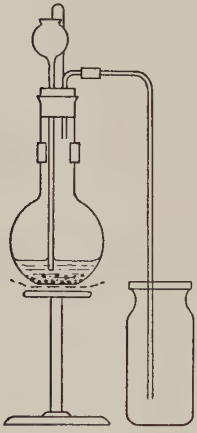

FIG. 33

b. Set up an apparatus as shown in Fig. 33. The 750-milliliter Florence flask serves as a generator. Fit it with a two-hole rubber stopper bearing a thistle tube and a double right-angled delivery tube with arms of unequal length. Collect the chlorine in bottles by the upward displacement of air. Three clean and dry bottles should be available.

Place in the flask 30 g. of sodium chloride (weigh this on the platform balance). Add 20 g. of manganese dioxide. Mix well, and replace the stopper, making sure that the lower end of the thistle tube is near the bottom of the flask. Pour through the thistle tube 50 ml. of concentrated sulfuric acid. Warm the flask to start the action and fill the bottles with the chlorine which comes off. Write the equation. Cover each bottle with a glass plate and save for use in the experiments on the properties of chlorine. The color of the gas against a white background enables one to tell when a bottle is full. To avoid inhaling the gas, keep the window of the hood closed, except when changing bottles. Also saturate about 200 ml. of distilled water with chlorine and save the solution (*stopper*). This may be done by admitting chlorine to a stoppered flask and shaking the flask to aid the solution of the gas. The stopper should be removed once or twice to allow the escape of the inclosed air.

c. An optional method for preparing chlorine is to substitute a dropping funnel for the thistle tube (Fig. 33). Place in the flask 15 g. of potassium permanganate and drop upon it concentrated hydrochloric acid from the dropping funnel. Regulate the flow of acid to give a steady flow of chlorine into the bottles. Write the equation.

48. Properties of Chlorine. *a.* Sprinkle finely powdered antimony into one of the bottles of chlorine. Antimony trichloride ($SbCl_3$) is formed. Write the equation. Does the antimony burn?

b. Twist together six 1-inch strands of copper wool. Heat these to redness and plunge them into a bottle of chlorine. Note the color of the fumes, add 3 ml. of water, and observe the color of the solution formed. Write the equation for the action. Is it combustion?

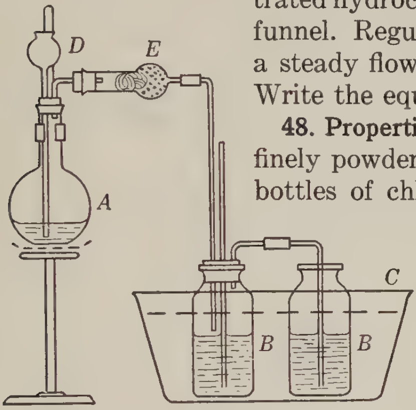

FIG. 34

c. Test the action of chlorine on strips of colored calico cloth when moist and when dry. Conclusions?

d. Add some of the solution of chlorine to a potassium iodide solution. What result is observed? Make a similar test with a bromide solution. What is the result? Write all equations.

49. Preparation of Hydrogen Chloride. Set up an apparatus as shown in Fig. 34. The parts are as follows: *A*, 750-milliliter Florence flask; *B*, wide-mouthed absorption bottles; *C*, pan of cold water; *D*, delivery tube; *E*, drying tube with glass wool and calcium chloride.

Study the diagram of the apparatus and decide on the purpose of the parts. Carefully assemble the apparatus. Place 25 g. of dry rock salt in the flask. Pour 100 ml. of water into each absorption bottle. Adjust the inlet tube of the first absorption bottle so that it reaches to within half an inch of the surface of the water. Why should it not dip into the water?

Pour 50 ml. of concentrated sulfuric acid through the thistle tube into the flask. Adjust the thistle tube so that it dips below the surface of the acid. Watch the action carefully to see that the foam does not rise and threaten to go up into the neck of the flask. As rapidly as seems safe, add more sulfuric acid. Note the exact amounts. When the action slackens, apply heat very cautiously to the flask, and finally heat it strongly until nearly all the salt disappears and effervescence has ceased. Do not heat until sulfur trioxide is formed and driven over. Discontinue heating as soon as the action appears complete and before any white fumes appear.

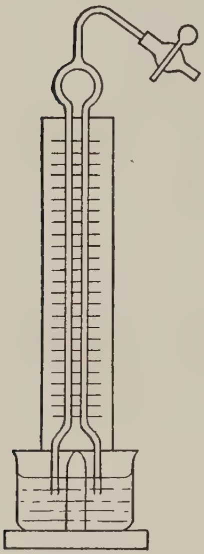

Fig. 35

The contents of the flask are very hot and must not be poured out, least of all into water. Let the contents cool thoroughly, then cautiously add cold water to the flask, and allow it to stand until the next laboratory period. Add more water if the solution is not complete.

Most of the hydrogen chloride has been dissolved in the first bottle (unless the latter was allowed to get too warm), and any remainder has been caught in the second bottle. To find the concentration of the acid in the first bottle, determine the specific gravity, using the apparatus shown in Fig. 35. The scale at the back is marked off in divisions of one-tenth inch. The acid is placed in the dish at the left and the water in the dish at the right, both being at the same temperature. The level is adjusted in both dishes so that the surface of each liquid is exactly at the zero point of the scale. Suction is applied at the top until the acid rises exactly to the 100 mark, when the pinchcock is closed. The reading of the scale at the top of the water column divided by 100 gives the specific gravity of the acid.

Look up in a specific-gravity table (Reference) the normality corresponding to the specific gravity found, measure the total volume of the acid, and compute the number of grams of HCl obtained in the first bottle. If a satisfactory yield cannot be accounted for, determine in the same way the acid in the second bottle. The contents of this, however, are not to be saved. Preserve the acid from the first bottle in a 250-milliliter narrow-necked glass-stoppered bottle.

How much of an excess of sulfuric acid was used in this preparation? What is the advantage of using such an excess?

50. Properties of Hydrochloric Acid. When hydrogen chloride is dissolved in water, the solution is called hydrochloric acid. Test the acid you have prepared as follows: (*a*) Dilute 1 ml. to 100 ml. and note the taste. (*b*) Test the action of the acid with red and with blue litmus paper. (*c*) Try its action on two metals of your own selection. What are the results? (*d*) Dissolve a chip of marble in the acid. Dilute and filter the solution if it is not clear. Evaporate the liquid and note the character of the residue. What is it? Give the equation.

EXERCISES

1. How many grams of chlorine will be produced by the action of hydrochloric acid on 800 g. of manganese dioxide?

2. How many liters of chlorine under standard conditions will be produced by the action of hydrochloric acid on 600 g. of lead dioxide?

3. What compounds are formed when phosphorus trichloride acts on water? Write the equation for this action.

4. Write the equations showing what is formed when hydrochloric acid acts (*a*) on metallic aluminum; (*b*) on zinc carbonate; (*c*) on ferric hydroxide; (*d*) on barium oxide.

5. What volume of hydrogen chloride gas measured at 21° and a pressure of 720 mm. can be obtained from 250 g. of rock salt, 90 per cent pure?

CHAPTER XI

THE HALOGEN FAMILY

51. Preparation and Properties of Bromine. (*Be very careful in handling bromine, as it inflicts very serious injury when brought in contact with the flesh.*) *a.* Obtain a small tubulated retort *A* and arrange it as shown in Fig. 36. The test-tube receiver *B* has in it water enough to cover the lower end of the retort. Place in the retort a mixture of 4 g. of sodium bromide and 3 g. of manganese dioxide. Clamp the retort in position and add to it, through a funnel placed in the tubulation, 15 ml. of cold dilute (1 to 2) sulfuric acid. Heat the retort very gently and note the collection of bromine at the bottom of the test tube. Continue the heating until all the bromine is driven over. Write the equation for the reaction.

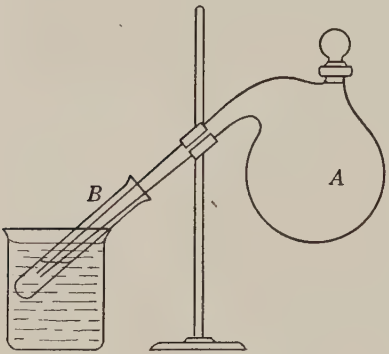

FIG. 36

b. Does any of the bromine dissolve in the water? How do you know? Add more water to the tube. What result is observed?

c. Test the bleaching action of the bromine solution on strips of colored calico cloth. Is it as good a bleaching agent as chlorine is?

d. Test the bromine solution with potassium iodide starch paper. Explain the result.

e. Add to a small portion of the solution some carbon disulfide and shake it vigorously. What color is observed? Tell which is the better solvent for bromine: water or carbon disulfide.

Make a similar test with chloroform and with ether. Which of the solvents tried is the best for bromine? Which is the poorest?

52. Preparation of Hydrobromic Acid. *a.* Place a small amount of sodium bromide in a test tube and add a few milliliters of concentrated sulfuric acid. Warm very gently. What is the color of the gas evolved? Blow your breath cautiously across the top of the test tube. Explain the result. Why is this not a good method for preparing hydrogen bromide? Write the equations.

b. Repeat, using phosphoric acid instead of sulfuric acid. Carry out the same tests. Note and explain any differences in the results. Write the equation.

c. Hydrobromic acid may be prepared in large quantities by the interaction of red phosphorus, bromine, and water. The phosphorus and bromine interact to form phosphorus tribromide. This in turn acts with water to form phosphorous acid and hydrogen bromide. Write equations for these changes.

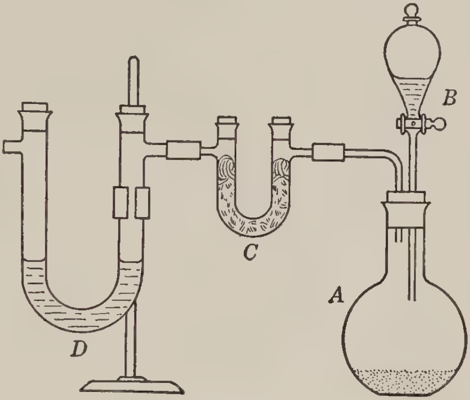

FIG. 37

The apparatus needed is shown in Fig. 37. Fit a 750-milliliter Florence flask *A* with a two-hole rubber stopper bearing a dropping funnel *B* and a right-angled delivery tube. Attach a small U-tube *C* containing a paste made by adding water to a mixture of red phosphorus and glass. This mixture is held in place by pads of glass wool. Connect the U-tube by a delivery tube and rubber stopper with a large U-tube *D* about one-fourth full of distilled water. Place in the flask *A* 7 g. of red phosphorus and 15 g. of sand. Add about 10 ml. of water. Assemble the apparatus (*HOOD!*), making sure that the stopcock fits tightly and turns readily. Then close the stopcock and pour about 12 ml. of liquid bromine into the funnel (*CARE!*).

Now allow a single drop of bromine to flow into the flask. Take care that bromine does not leak out round the stopcock and reach your fingers. Add the bromine drop by drop as slowly as seems advisable. Note the absorption of hydrogen bromide in the large U-tube. When the action is complete, pour the hydrobromic acid into a graduate, measure, and record the volume of the acid. Preserve it in a glass-stoppered bottle. Wash out the generating apparatus thoroughly under the hood.

53. Properties of Hydrobromic Acid. *a.* Test the action of separate portions of the solution from Experiment 52, *c*, on sodium carbonate, on sodium hydroxide solution to which two drops of phenolphthalein have been added, and on zinc. Write the equations for the chemical actions noted.

b. Treat about 0.1 g. of manganese dioxide with 5 ml. of the solution. Extract the product with ether. What is it? Account for its formation.

54. Preparation of Barium Bromide ($BaBr_2 \cdot 2H_2O$). Weigh out about 4 g. of barium carbonate ($BaCO_3$). Place it in a 100-milliliter beaker and cover it with a watch glass. Lift the cover and add cautiously a few milliliters of the hydrobromic acid prepared in the preceding experiment. Replace the cover quickly. When the effervescence ceases, stir the mixture with a glass rod, and then repeat the addition of hydrobromic acid. Continue the addition until there is a slight excess of acid. Now stir the mixture vigorously. Boil the contents of the beaker carefully for about five minutes. Filter quickly and evaporate the filtrate to about two thirds of its original volume. Allow the solution to cool slowly. If no crystals form, evaporate the solution to half its original volume. Separate the crystals and evaporate the mother liquor. Repeat the evaporation and crystallization if it seems advisable. Dry the crystals between filter papers and weigh them. Preserve them in a bottle for inspection.

55. Properties of Bromides. *a.* Dissolve one fourth of your barium bromide in 4 ml. of water. Add a few drops of a dilute silver nitrate solution to 2 ml. of the bromide solution. What is the color of the precipitate? Write the equation for its formation. Test the solubility of this precipitate in concentrated ammonium hydroxide.

b. Recall the action of chlorine water on bromides in solution. Repeat the test with 2 ml. of barium bromide solution. Then add 3 ml. of chloroform and shake the mixture. What change is noted? Does chloroform make the test more delicate?

56. Preparation of Iodine. Place a mixture of about 2 g. of potassium or sodium iodide and about 1 g. of manganese dioxide in a deep evaporating dish. Pour into the dish 4 ml. of concentrated sulfuric acid. Cover the dish with a watch glass somewhat larger in diameter than the dish. The convex side of the watch glass is placed downward. Fill the concave side of the watch glass nearly full of cold water. Apply a very gentle heat to the evaporating dish. When dense violet fumes are seen rising from the mixture in the dish, remove the flame. After four or five minutes remove the watch glass and observe the crystals of iodine deposited on its lower side. Scrape them off for further study. Write the equation for the production of iodine by this method.

57. Properties of Iodine. *a.* Place a few crystals of iodine in a test tube and warm it carefully in a low flame. What color do you observe in the test tube? Do the vapors escape into the air? Note the deposit which forms on the cool upper portion of the test tube. Account for this phenomenon.

b. Test the solubility of the iodine crystals in water, in chloroform, in carbon disulfide, in alcohol, in ether, and in a water solution of potassium iodide. Tabulate the colors observed. In which solvent does iodine seem to dissolve the best?

c. Test the effect of iodine on freshly prepared starch paste by adding a few drops of iodine dissolved in potassium iodide to water containing a little starch paste. What is the color? Heat the test tube carefully. Does the color disappear? Cool the test tube quickly under the tap. What result is observed? Repeat the test and the heating, but now allow the tube to cool slowly in the air. What is the result?

58. Preparation of Hydriodic Acid. *a.* Repeat Experiment 52, *a*, substituting sodium iodide for sodium bromide. Note carefully all results. Write the equations.

b. Repeat Experiment 52, *b*, substituting sodium iodide for sodium bromide. Write the equation for the action.

c. Hydrogen iodide may be prepared by a similar method to that by which hydrogen bromide was prepared in Experiment 52, *c.* To illustrate this on a very small scale, place 0.1 g. of red phosphorus in a test tube, moisten it with water, and then drop a crystal of iodine into the tube. Warm the mixture gently. Blow your breath across the top of the test tube. What result is noted? Write the equation for the preparation of hydrogen iodide by this method.

d. Weigh out 5 g. of iodine and powder it finely by grinding in a mortar. Place it in a tightly stoppered flask or test tube ready for use. Pour 25 ml. of distilled water into a 100-milliliter Erlenmeyer flask and add to it about 0.5 g. of iodine. Fit the flask with a one-hole rubber stopper bearing a glass tube. The lower end of the tube should be just above the level of the water. Attach the upper end of the tube to the exit tube of a hydrogen-sulfide generator (*HOOD!*) or to the hydrogen-sulfide line in the laboratory. Admit hydrogen sulfide into the flask, loosening the rubber stopper momentarily until the air is displaced by hydrogen sulfide. Then stopper the flask tightly and allow the solution to absorb hydrogen sulfide under the pressure of the generating system (*CARE!*). When the brown color of the solution has disappeared, shut off the supply of hydrogen sulfide and introduce about half a gram more of iodine. Again absorb hydrogen sulfide until the solution is nearly colorless. Repeat these half-gram additions until about 3 g. have been used up. Add the remaining 2 g. and allow the flask to stand, with repeated shakings, until all the iodine is dissolved. Again absorb hydrogen sulfide until the solution is nearly decolorized. Then pour the solution into another flask, holding back the clotted lumps of sulfur with a stirring rod. Pass carbon dioxide through the solution until all the excess hydrogen sulfide is carried off by the dioxide. Shake the flask to aid in the flocculation of the suspended sulfur. Filter the solution to rid it of the sulfur. The liquid is now a very dilute aqueous solution of hydriodic acid. It may be tested as it is to establish its identity.

The solution may be concentrated by fractional distillation. The solution is placed in a distilling flask fitted with a thermometer and an inlet tube for hydrogen (Fig. 38). The side arm of the distilling flask is connected with a Liebig condenser and this in turn with a receiving flask tightly stoppered save for an outlet tube. The apparatus is filled with hydrogen, and hydrogen is kept passing slowly through it during the distillation.

The first portion of the distillate is nearly pure water, and the thermometer registers only a little, if any, above 100°. When the thermometer registers 105°, the receiving flask is changed; when the thermometer reads 120°, the flask is again changed. Nearly all the remaining part of the solution distills over at about 126°, and all that comes over in this range is collected for use. What is the approximate composition of the acid?

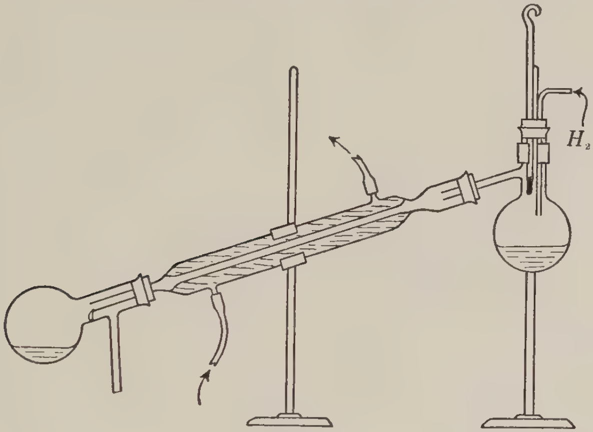

FIG. 38

59. Preparation of Sodium Iodide. Place 2 ml. of sodium hydroxide solution in a small beaker and add slowly small amounts of hydriodic acid. Test the solution from time to time by removing a drop with a stirring rod and placing it on a piece of blue litmus. Stir and test the solution after each addition of acid. When the solution is just acid, as shown by its effect on the litmus paper, enough acid has been added. Save this solution for the following experiment.

60. Properties of Iodides. *a.* Test 2 ml. of the solution from Experiment 59 with silver nitrate solution. Write the equation for the action. What is the color of the precipitate? Is it soluble in concentrated ammonium hydroxide? Compare its solubility with that of the silver chloride precipitate; with that of the silver bromide precipitate.

b. Add chlorine water to the remainder of the solution. Shake with chloroform. Explain the results observed.

EXERCISES

1. Devise a table showing the physical properties of the halogens.
2. Devise a similar table showing the chemical properties of the halogens.
3. What weight of hydrogen bromide can be prepared by the action of excess bromine and water on 200 g. of red phosphorus?
4. How is hydrofluoric acid prepared?
5. How is fluorine prepared?
6. How is hydrofluoric acid stored or handled?
7. Are iodides essential factors in animal diets? Why?
8. Discuss briefly the usual sources of iodine in the diet.
9. What are the usual commercial sources of iodine?
10. Discuss the occurrence of bromine in nature.
11. What is the medicinal use of bromides?
12. How might one test for starch in plant or animal tissues?

CHAPTER XII

CALCULATIONS

61. Weight Relationships in Chemical Change. Tabulate all data. *a.* Weigh a porcelain crucible (be sure that it is clean and dry). Place about 1 g. of sodium hydrogen carbonate (*storeroom*) in the crucible and weigh accurately. Place the crucible on a pipestem triangle supported on a ring stand and heat gently with a low flame of a Bunsen burner. Continue the heating for about ten minutes. Then gradually increase the height of the flame and heat strongly for about fifteen minutes. Allow the crucible to cool and then weigh it. Heat again for about five minutes and reweigh the cooled crucible. Continue until constant weight is reached. The material remaining in the crucible is sodium carbonate. Carbon dioxide and water were driven off during the heating. Write the equation for this chemical action.

Calculate the theoretical weight change according to the equation and compare it with the actual change in weight. Calculate the per cent of error. What are the possible sources of error in this experiment? Save the crucible and contents for part *b*.

b. Select a small watch glass which will cover the crucible from *a* completely. Find its weight. Pour 2 ml. of water into the crucible. Pour four or five *drops* of dilute hydrochloric acid down the side of the crucible and cover it with the watch glass. Note the effervescence due to the escape of bubbles of carbon dioxide gas. Write the equation. When the bubbling has ceased, add a few drops more of the dilute acid. Allow the effervescence to subside and repeat the addition. Continue until further addition of acid causes no bubbling. Then add 2 ml. of concentrated hydrochloric acid to insure complete reaction. Lift the watch glass and, using distilled water, rinse back into the crucible any material on the underside of the watch glass. Lay the watch glass aside on a clean piece of paper and, very carefully and slowly, evaporate the solution on the water bath. Cool the crucible and watch glass, dry the outside of them, and weigh them. Examine the residue in the crucible. Enumerate its physical properties. Taste it. What is it? Write the equation. How much of this material could be made had you used 10 g. of sodium bicarbonate? What would the theoretical yield be? Why the difference?

EXERCISES

1. Calculate the per cent of each of the elements in (*a*) $CaCO_3$, (*b*) $ZnSO_4$, (*c*) $Ba_3(PO_4)_2$, (*d*) $PbCO_3$, and (*e*) $CaSO_4 \cdot 2\ H_2O$.

2. What weight of metallic zinc will be needed to prepare 100.8 g. of hydrogen?

3. What weight of cupric oxide will be formed when 1000 lb. of copper is oxidized?

4. What weight of copper will be formed when 2000 lb. of cuprous oxide is reduced by hydrogen?

5. What weight of ferric oxide can be formed from 200 g. of pure iron wire?

6. What weight of hydrogen must be burned to form 200 g. of water? What weight of oxygen will be needed?

7. What corrected volume of hydrogen must be burned to give 150 g. of water?

8. What weight of carbon dioxide will be formed when 144 g. of carbon is burned in air?

9. What volume of hydrogen chloride measured at 0° and 760 mm. may be prepared by the action of 64.949 g. of sodium chloride with an excess of sulfuric acid?

$$NaCl + H_2SO_4 \longrightarrow NaHSO_4 + HCl$$

Assume the reaction to be 90 per cent complete.

10. Calculate the weight of hydrogen which will be formed when 3 g. of aluminum reacts with dilute hydrochloric acid (HCl).

11. What weight of quicklime (CaO) must be slaked to give 600 lb. of slaked lime ($Ca(OH)_2$)? What weight of water will be required?

12. How many pounds of hydrogen can be obtained when 326.85 lb. of zinc is acted on by hydrochloric acid?

$$Zn + 2\,HCl \longrightarrow ZnCl_2 + H_2$$

13. A volume of gas was kept under a constant pressure. When heated from 10° to 12° its volume change was 10 ml. What was the original volume of the gas?

14. On a day when the barometric pressure was 730 mm. a balloon had the power of just lifting 2 metric tons. What weight would the same balloon have been capable of lifting had the atmospheric pressure been normal?

15. Oxygen is diatomic; 3 volumes of it change into 2 volumes of ozone. Prove that a molecule of ozone is triatomic.

16. The molecular weight of hydrogen chloride is 36.465. Calculate the following approximate values: (*a*) normal density; (*b*) absolute density; (*c*) density compared with that of air.

17. Calculate the equivalent weight of aluminum from the following experimental data: 1.95 g. of the powdered metal when treated with sodium hydroxide gave 2.615 liters of hydrogen, measured over water at 17° and at a pressure of 758 mm.

CHAPTER XIII

OXYGEN COMPOUNDS OF THE HALOGENS

62. Preparation and Properties of Bleaching Powder. *a.* Place about 10 g. of freshly slaked lime in a 500-milliliter flask fitted with a one-hole rubber stopper bearing a glass delivery tube. Set up a chlorine generator and attach the flask to the exit tube. Open the stopcock, allow the chlorine to displace the air, and then stopper the flask tightly and allow the lime to absorb the chlorine. Write the equation for the chemical action. Allow the action to proceed for about thirty minutes, then remove the flask, and stopper it tightly with a cork. A cylinder of chlorine may be used, provided the necessary precautions are taken. What are they?

b. Take about 8 g. of the material prepared in *a* and stir it into 40 ml. of water. Filter and wash the residue once with distilled water. Allow the washings to fall into the filtrate. Use the filtrate for the following tests.

c. Add to 4 ml. of the solution a few drops of dilute hydrochloric acid. Note the increase in the odor. Write the equation. Blow carbon dioxide from the breath through another 4-milliliter portion. What result is observed? Write the equation.

d. Using strips of colored calico cloth, test the bleaching action of the solution from *b*, first as obtained, secondly when dilute acid is added, and thirdly when a base is added. What do you conclude as to the best conditions for bleaching?

63. Preparation of Sodium Hypochlorite. Prepare 25 ml. of a 10-per-cent solution of sodium hydroxide (assume that solid sodium hydroxide has a purity of 90 per cent). Add enough anhydrous sodium carbonate to make the solution 2.5 per cent with respect to this salt. Introduce the resultant solution into a 50-milliliter Erlenmeyer flask provided with a two-hole rubber stopper. The stopper should carry a delivery tube which reaches to the bottom of the flask, and an outlet tube which should be bent so as to dip into a concentrated solution of sodium hydroxide (Fig. 39). Weigh the flask and its contents. Connect the apparatus with a chlorine tank (or generator). Allow chlorine to enter the flask at such a rate as to allow but little to escape through the outlet tube. Permit enough chlorine to enter so that the weight of the flask and contents increases 2.2 g. In order not to exceed this amount, weigh the flask and the solution from time to time. When the proper amount of chlorine has been absorbed, preserve the solution for use in the following experiment.

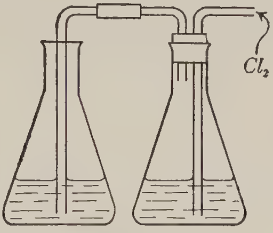

FIG. 39

64. Properties of Sodium Hypochlorite. *a.* Immerse strips of differently colored calico in 5 ml. of this solution. Observe whether the colors are bleached or not. Remove the strips and add new ones of the same material. Make the solution slightly acid by the addition of dilute sulfuric acid. Allow the strips to remain in the mixture for about a minute, remove, and wash them thoroughly with water. Which is the more effective: the hypochlorite alone or the hypochlorite and the acid?

b. Test the hypochlorite solution with potassium iodide starch paper. In the same way test the solution after the addition of acid. What result is observed? Give the equation.

c. Treat 2 ml. of chromium sulfate solution with an equal amount of ammonium hydroxide. Filter off the precipitate, remove it from the filter paper, and heat it in a test tube with 5 ml. of the hypochlorite solution. Compare the color of the resulting solution with that of the solution of potassium chromate in the laboratory. After making this comparison, give the name of the product.

65. Preparation of Potassium Chlorate. Dissolve 2 g. of solid potassium hydroxide and 5 g. of solid sodium hydroxide in about 10 ml. of water in a 100-milliliter Erlenmeyer flask. The solution may appear somewhat murky. Fit the flask with a two-hole rubber stopper bearing a glass delivery tube and an exit tube bent at right angles to dip into a dilute (6-*N*) solution of sodium hydroxide. Now pass chlorine into the flask, allowing the reaction to heat up the solution. Shake the flask gently from time to time. When the solution is saturated with chlorine, remove the generator connections, boil the solution very gently to remove the excess chlorine, and filter on a suction funnel while still hot. Before all the liquid has drained through the filter, wash out the flask twice with 10 ml. of boiling water and pour the rinsings through the filter. Cool the filtrate to 0° (*freezing-mixture*) and collect the potassium chlorate crystals which are formed. Dissolve these crystals in three times their weight of water and recrystallize them. Dry between filter papers. Submit your preparation to your instructor.

66. Preparation of Potassium Bromate. Dissolve about 4 g. of solid potassium hydroxide in 10 ml. of water in a 50-milliliter Erlenmeyer flask. Cool this solution to room temperature and take it to the hood. Measure out in a graduate about 1.5 ml. of liquid bromine (*DANGER!*). Add this, in five separate portions, to the potassium hydroxide solution. After each addition rotate the flask until the color of the bromine has disappeared before adding more bromine. When all the bromine has been added, heat the contents of the flask until the excess bromine is expelled, then cool to about 10° (*ice*). Filter out the crystals. What is present in the filtrate? Redissolve the crystals in hot water, filter while hot, if necessary, and allow the filtrate to cool to 10°. Collect the crystals as before. Dry the crystals by pressing them between filter papers.

67. Preparation of Potassium Iodate and Properties of Iodates. Dissolve 3 g. of potassium chlorate in about 20 ml. of water in a 50-milliliter flask. Powder about 3 g. of iodine in a mortar and add it to the solution. Place a funnel in the neck of the flask and add to the mixture 1 ml. of dilute (6-N) nitric acid. Warm gently until the violet fumes of iodine nearly fill the flask. Then remove the flame and watch the action closely. If iodine vapors start to escape through the funnel, slow down the action by placing the flask in a pan of cold water. When the reaction seems complete, warm the solution until the excess iodine has disappeared (note the color). Then boil for about ten minutes to drive off the excess chlorine. Now add slowly a dilute solution of potassium hydroxide until the solution turns red litmus blue. Test for this by dipping a stirring rod into the solution and touching it to litmus paper. Allow the solution to cool, and recover the crystals of potassium iodate which separate. Redissolve the crystals and again crystallize. Save the product for inspection.

Dissolve some potassium iodate in water. Add a dilute solution of silver nitrate. Filter out the precipitate, and test its solubility in concentrated ammonium hydroxide. Make this solution just acid with nitric acid. Filter and wash. Now heat the precipitate with sodium hydrogen sulfite for three minutes. Test the solubility of the residue in concentrated ammonium hydroxide. What is the residue?

EXERCISES

1. What weight of potassium bromate can be formed by the action of 20 g. of bromine on an excess of potassium hydroxide?

2. Why was a mixture of sodium hydroxide and potassium hydroxide used in making potassium chlorate in Experiment 65?

3. Make a table showing the known oxides and oxygen acids of the halogens.

4. Will chlorine displace iodine from all its compounds?

5. Which of the halogens is the most active as a nonmetal?

6. Which should you expect to be the more vigorous agent in oxidizing hydrogen chloride: chloric acid or iodic acid?

CHAPTER XIV

SOLUTION

68. Rate of Solution. Select three crystals of cupric sulfate of equal size. Place the first two in separate test tubes and fill the tubes about one third full of water. Allow one tube to remain undisturbed, and shake the other vigorously at frequent intervals. Meanwhile powder the third crystal and place the powder in another test tube about one third full of water. In which tube does the salt dissolve most rapidly? What is the effect of agitation on the rate of solution? What is the effect of the state of division on the rate of solution? With a new crystal of cupric sulfate, try the effect of heat on the rate of solution. What are the results? What is your conclusion?

69. Diffusion. Dissolve a small piece of potassium permanganate in a test tube of water. Fill your large Florence flask nearly full of water and add to it the solution from the test tube. Shake the flask well and observe the color of the solution. Is the color the same in all parts of the flask? Why? Does the solute settle out? Why? Place a crystal of potassium permanganate in a test tube of water, being careful not to shake it. Let the tube stand an hour, and then observe. Is the color the same in all parts? Explain. Is the rate of diffusion of the permanganate slow or rapid?

70. Solubility. *a.* Dissolve all the sodium chloride you can in 100 ml. of water. Keep adding salt and shaking the solution till some solid remains undissolved. Do the same with calcium sulfate. How do the amounts compare?

b. Dissolve all the potassium nitrate (saltpeter) possible in 5 ml. of hot water. Cool the solution under the faucet. What result is observed? Compare the solubility of potassium nitrate in hot water and in cold.

c. Test the solubility of roll sulfur in water and in carbon disulfide (CS_2). Test the solubility of iodine in alcohol, in carbon tetrachloride, and in potassium iodide (KI) solution. What results are observed?

71. Supersaturation. Place 30 ml. of distilled water in a 100-milliliter beaker. Heat the water to a temperature just below boiling. Add solid sodium thiosulfate ($Na_2S_2O_3$) until there is an undissolved residue. Keep the solution hot for five minutes. Then pour 20 ml. of the clear hot solution into a warm 50-milliliter Erlenmeyer flask. Cork this flask and place it aside to cool. Do not move or jar it while it is cooling. After an hour inspect the contents of the flask. What do you observe? Then add to the solution a crystal of the sodium thiosulfate. Account for the observed result.

72. Determination of the Solubility of Ammonium Chloride. The class should be divided into equal sections. The solubility of ammonium chloride is to be determined at each 10° interval from 0° to 90°, each student making the determination at *one assigned* temperature. The values obtained by each section will then be assembled to show the solubility curve of ammonium chloride.

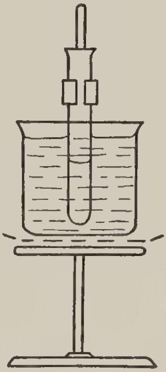
FIG. 40

Clamp a large test tube in a beaker of water as shown in Fig. 40. Place 15 ml. of water in the test tube and introduce powdered ammonium chloride. Stir the mixture with a glass rod. Place a thermometer in the beaker and then raise or lower the temperature to that assigned. Maintain it at the assigned point, stirring the water with the thermometer. Add more powdered chloride until there is an undissolved residue. Hold the temperature at the desired point for at least fifteen minutes. Weigh a small evaporating dish. Pour into it, with the aid of a glass rod, about 5 ml. of the clear solution of ammonium chloride. Weigh the dish and contents and evaporate the liquid to dryness on a water bath. Weigh them again. From these weighings calculate the amount of the salt that would be contained in 100 g. of the solution at the observed temperature. Draw up a tabular form of record showing the weight dissolved in 100 g. of the solution at each of the assigned temperatures. Include a column for the name of the observer. Plot these values on coördinate paper (see the instructor).

73. Heat of Solution. Prepare a calorimeter as represented in Fig. 41. The calorimeter beaker *A* should hold approximately 400 ml., and the larger beaker *B* should hold at least 1 liter. Provide three flat corks of equal thickness to serve as supports for *A*, a stirrer *C* made from a piece of light-weight rod and bent into a ring at the end, and a thermometer *D*. Cut a cardboard cover *E* circular in form, to fit over *A* and inside *B*; also a cover *F* for *B*, which may be left square. In the middle of each cover punch a hole, through which the thermometer may be pushed; make it small enough to hold the latter in place. A second hole must also be punched in each cover to provide for the handle of the stirrer.

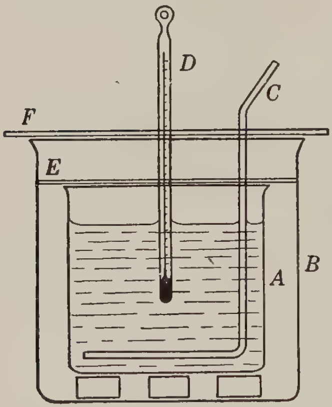

FIG. 41

Accurately measure 300 ml. of distilled water and pour it into the calorimeter beaker *A*. Weigh out 15 g. of ammonium chloride and place it in a test tube. Immerse the lower end of the tube in the water for about five minutes. Read and record the temperature of the water. Remove the tube with as little loss of water as possible, dry it quickly, and at once pour the chloride into the water in the calorimeter beaker *A*. Assemble the apparatus and stir the water thoroughly, using a steady and regular motion. The stirrer must not be allowed to splash out any of the water. Note the change in temperature and read the lowest temperature reached.

Knowing the temperature change and the weight of the water, calculate the thermal effect accompanying the solution of the salt. Some heat is always absorbed from the beaker etc.; experience shows that for the sizes specified this will be about 12 cal. Add this value in your calculations. Calculate the heat of solution of 1 g. of ammonium chloride and of a gram-molecular weight.

EXERCISES

1. Explain, in terms of the kinetic molecular theory, the effect of the state of division on the rate of solution. Explain in a similar fashion the effect of increased temperature on the rate of solution.

2. Why will a solid solute supported just below the surface of a liquid solvent dissolve sooner than if placed at the bottom of the containing vessel?

3. How can one distinguish experimentally between a saturated, a supersaturated, and an unsaturated solution?

CHAPTER XV

CHEMICAL PROPERTIES OF ACIDS, BASES, AND SALTS

74. General Properties of Acids. *a.* From your text and laboratory notes make a summary of the common properties of acids, such as their action on metals, bases, oxides of metals, litmus, etc. Verify your statements, using hydrochloric acid.

b. Repeat these experiments, using acetic acid. Do you observe any difference when acetic acid is used instead of hydrochloric acid? Note particularly the rates of reaction of these acids, using with each a piece of magnesium ribbon one inch in length.

c. Try the action of concentrated nitric acid and of concentrated hydrochloric acid on strips of copper. From your observations do you consider that this experiment illustrates a general property of acids? If you are not convinced, heat strips of copper with sulfuric acid (1 part concentrated acid to 1 part of water).

d. Treat separately small samples of sodium carbonate (Na_2CO_3) with dilute solutions of each of the four acids mentioned in this experiment. Introduce a burning splint into the gas formed. Do the results show that all these acids behave alike with the carbonate?

75. Preparation of Sodium Hydroxide. To 10 g. of sodium carbonate and 6 g. of quicklime in a 150-milliliter beaker, add 95 ml. of water. Heat the mixture to 80° and keep it at this temperature until the action is complete. During the heating, the liquid should be stirred continuously. The reaction is at an end when a small portion of the filtered solution gives no evolution of carbon dioxide when treated with dilute sulfuric acid. Test a few drops of the solution after each ten-minute period of heating, to determine whether or not the reaction is complete. When your test shows no carbon dioxide, filter the solution, concentrate the filtrate to half its original volume, and save the solution for the next two experiments.

76. General Properties of Bases. Place a drop of the sodium hydroxide, just prepared, on a strip of red litmus. What is the change? Note the feel of the hydroxide by rubbing a drop or two between the index finger and the thumb. Dilute from 1 ml. to 10 ml. and taste the resulting solution. What is the taste? Test potassium hydroxide, calcium hydroxide, and ammonium hydroxide in the same way, and compare results. Note the formula of each base. What group of atoms (radical) is common to bases?

77. Neutralization. Pour 2 ml. of the sodium hydroxide into an evaporating dish. Add an equal amount of dilute (6-molar) hydrochloric acid. Test the solution with both red and blue litmus by touching the liquid with litmus papers. If the red turns blue, add a few drops more of the acid; if the blue turns red, add a few drops more of the base. Continue these additions and testings until neither color of litmus is changed. Then carefully evaporate the solution almost to dryness. Let it cool, and examine the solid which crystallizes out. What is the taste of the product? What is it? Give the equation for its formation.

78. Properties of Salts. Dissolve 1 g. each of the following salts in separate test tubes, using 10 ml. of water in each: potassium nitrate (KNO_3), sodium carbonate (Na_2CO_3), sodium hydrogen carbonate ($NaHCO_3$), sodium chloride ($NaCl$), cupric sulfate ($CuSO_4$), and aluminum sulfate ($Al_2(SO_4)_3$). Test each solution with blue litmus and with red. Tabulate your results. Do these tests show that salts possess any common property? Should you conclude from these experiments that salts contain a common radical? From the formation of sodium chloride (a salt) by neutralization, what do you conclude as to the composition of salts?

EXERCISES

1. Calculate the amount of pure calcium hydroxide which is necessary to react with 10 g. of sodium carbonate. What impurities are likely to be present in sodium hydroxide prepared from these substances.

2. What materials are used in preparing sodium hydroxide electrolytically? What other products are formed?

3. Barium sulfate does not dissolve to an appreciable extent in water. Suggest a way of preparing the salt.

4. Sodium chlorate ($NaClO_3$) contains chlorine, but does not give a precipitate with a dilute solution of silver nitrate. Sodium chloride does give a precipitate with silver nitrate. How can you harmonize these two statements?

5. Write equations showing the salts formed by neutralizing each of the acids named with each of the bases named:

ACIDS	BASES
Sulfuric acid (H_2SO_4)	Potassium hydroxide (KOH)
Nitric acid (HNO_3)	Calcium hydroxide ($Ca(OH)_2$)
Phosphoric acid (H_3PO_4)	Ammonium hydroxide (NH_4OH)
Acetic acid ($HC_2H_3O_2$)	Barium hydroxide ($Ba(OH)_2$)

CHAPTER XVI

EQUILIBRIUM AND THE SPEED OF CHEMICAL REACTIONS

79. Reversible Reactions. *a.* Prepare 10 ml. of a saturated solution of sodium hydrogen sulfate ($NaHSO_4$). Add to this solution an equal volume of concentrated hydrochloric acid. Allow the precipitate to settle and pour off the supernatant liquid. Wash the crystals by three or four decantations, using dilute hydrochloric acid. Place the solid in an evaporating dish and heat it until it is dry. Examine the product with the aid of a lens. Taste it. Dissolve a few crystals in distilled water and treat the solution with a drop of silver nitrate. What is the result? What is the name of the salt obtained by the action of concentrated hydrochloric acid on the solution of sodium hydrogen sulfate? Compare the result of the reaction with that of treating sodium chloride with concentrated sulfuric acid. Why is it possible that each of these opposite reactions may be made very nearly complete? Under what conditions would neither of them be nearly complete?

b. Place a few crystals of bismuth chloride ($BiCl_3$) in a test tube and add about 1 ml. of water. To this add concentrated hydrochloric acid, drop by drop and with constant stirring, until the solution clears up. Now pour the solution into a beaker of water. What are the results? Again add acid as before. What do you conclude about this action?

80. Chemical Equilibrium. To 200 ml. of water in a beaker add, first, 1 ml. of a ferric chloride solution, and then 1 ml. of a solution of ammonium thiocyanate. Note the red color of the solution of ferric thiocyanate. Pour 4 ml. of the solution into each of four test tubes. Save the first for comparison. To the second add 0.5 ml. of ammonium thiocyanate solution. Note any color change. To the third add 0.5 ml. of ferric chloride solution. Note any color change. Saturate the fourth with ammonium chloride, using the solid. Note the color change. Explain the color changes noticed in this experiment.

81. Effect of a Change in Temperature on the Speed of a Chemical Reaction. An increase in temperature of 10° approximately doubles the rate at which many reactions take place. This may be illustrated by noticing the time required for an iron nail to decolorize an acidified potassium permanganate solution at different temperatures. The concentration (see Appendix) and volume of the acidified permanganate solution must be the same.

Secure five bright zinc-coated or galvanized nails of the same size and kind. Measure into each of five test tubes exactly 15 ml. of the acidulated permanganate solution prepared for you. Place all these test tubes in a beaker of water and heat the water almost to boiling. Allow the tubes to stand in the beaker for five minutes so as to come to the temperature of the water. Notice and record the temperature of the purple solution. Drop a nail into one of the test tubes, and note the time required (in seconds) for the nail to decolorize the solution. When the temperature of the water has dropped about 10°, place a nail in the second test tube. Again allow the temperature to drop about 10°, and place a nail in another test tube. Use the other two test tubes at temperatures still lower. Meanwhile note the time required for the solution in each tube to become decolorized. Record these values in tabular form, showing the time required for bleaching at each temperature. Is there any simple relationship between these times?

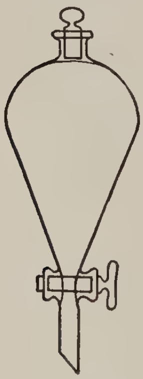

FIG. 42

82. Law of Partition. Introduce into a small separatory funnel (Fig. 42) 10 ml. of a saturated solution of bromine water. To the bromine water add an equal volume of carbon tetrachloride. Stopper the funnel and shake its contents vigorously for a minute or two. Allow the mixture to separate into two layers. Draw off the lower one and discard it. Allow the upper layer to run into a test tube. Repeat the experiment, using 5 ml. of carbon tetrachloride. Draw off the lower layer and then add another 5-milliliter portion of carbon tetrachloride to the funnel.

Shake the contents as before and remove the lower layer. Allow the upper layer to run into a test tube. Compare the intensity of color of the liquid in this test tube with that of the liquid in the first test tube. Explain any difference observed.

EXERCISES

1. Explain the reversible action noted in Experiment 79, *a*, in terms of the kinetic-molecular theory.

2. Explain the phenomena noted in Experiment 80 by means of the kinetic-molecular theory. Apply the law of molecular concentration.

3. Given the equation $2\,NO + O_2 \rightleftarrows 2\,NO_2 + \text{heat}$, explain the effect of a change in temperature and in pressure on the chemical action represented.

CHAPTER XVII

IONIZATION

83. Properties of Ions. *a.* Obtain a few small crystals of cupric sulfate ($CuSO_4 \cdot 5\,H_2O$), cupric chloride ($CuCl_2 \cdot 2\,H_2O$), and cupric nitrate ($Cu(NO_3)_2 \cdot 6\,H_2O$). Note their colors. Dissolve each salt separately in the smallest amount possible of water. Note the colors of the solutions. Dilute each solution ten times with distilled water. What color changes, if any, are seen? Obtain crystals of sodium sulfate ($Na_2SO_4 \cdot 10\,H_2O$), calcium chloride ($CaCl_2 \cdot 6\,H_2O$), and potassium nitrate (KNO_3), and dissolve each of them in water. What is the color of these solutions? What is the color of the sulfate ion (SO_4^{--})? of the chloride ion (Cl^-)? of the nitrate ion (NO_3^-)? of the sodium ion (Na^+)? of the cupric ion (Cu^{++})?

b. Obtain 0.2 g. of solid cupric bromide ($CuBr_2$). Add 1 ml. of water to it in a test tube. Continue to add water, a few drops at a time, until the tube is about half full. What changes in color take place? Does the degree of ionization increase with dilution or decrease?

To another portion of cupric bromide in a test tube add water cautiously until the solution is green. Divide the solution into three portions. To one add solid potassium bromide (KBr). What change in color occurs? To another add solid cupric chloride. Note the change in color. Save the other for comparison.

c. Mix solutions of sodium sulfate (Na_2SO_4) and barium chloride ($BaCl_2$). Is the action immediate? Why? Would the dry substances have interacted so quickly? Explain.

84. Degree of Ionization. The degree of ionization of an ionogen in solution is often measured by electrical conductivity. According to Arrhenius the conductivity at a given dilution is greater, the greater the degree of ionization at a given temperature.

Obtain two copper wires six inches long. Pass these through a cork (Fig. 43) so that about three inches of each protrude. Introduce the wires into a test tube of distilled water and connect them with the binding posts of the conductivity apparatus in the laboratory. Test the following solutions for their conductivity by noting the brightness of the lamp, care being taken to rinse the wires thoroughly with distilled water after each test:

a. A 5-per-cent sugar solution.
b. A 5-per-cent alcohol solution.
c. A 0.1-*N* solution of hydrochloric acid.
d. A 0.1-*N* solution of sulfuric acid.
e. A 0.1-*N* solution of nitric acid.
f. A 0.1-*N* solution of phosphoric acid.
g. A 0.1-*N* solution of acetic acid.
h. A 0.1-*N* solution of ammonium hydroxide.
i. A 0.1-*N* solution of sodium hydroxide.
j. A 0.1-*N* solution of sodium chloride.
k. A 0.1-*N* solution of sodium acetate.
l. A 0.1-*N* solution of mercuric chloride.
m. Pure toluene (use dry electrodes).
n. A solution of hydrogen chloride in toluene (use dry electrodes).

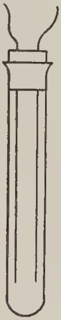

Fig. 43

RECORD

Solution	Nonconductor	Poor Conductor	Good Conductor

Which of these solutions should be classified as intermediate between a good conductor and a poor one?

85. Electrolysis of a Sodium Sulfate Solution. Fill a U-tube with a solution of sodium sulfate colored with neutral litmus. Introduce electrodes, from a source of direct current, into the arms of the tube as shown in Fig. 44. Allow the current to pass through the solution, and note the changes in color at the electrodes. To which electrode do the sulfate ions migrate? Upon what do you base your conclusion? What gases are given off?

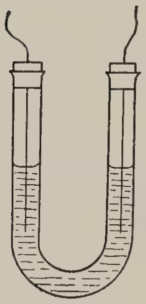
FIG. 44

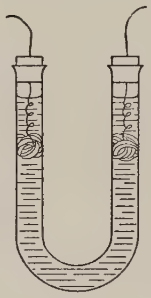
FIG. 45

86. Migration of Ions. Fill a U-tube two thirds full of 0.1-molar cupric sulfate. Introduce a plug of glass wool into each arm of the tube as shown in Fig. 45. Carefully fill each arm with dilute sulfuric acid and insert aluminum electrodes into the acid. Connect these with a source of direct current. Allow the current to pass through the solution for five minutes, noting the gradual change in color in each arm of the U-tube. Near which plug of glass wool does the color fade? Toward which electrode do the cupric ions migrate?

EXERCISES

1. What would be the difference in the boiling-points of a 0.1-molar solution of sugar and of sodium chloride?

2. How many faradays would be required to liberate 250 g. of copper from cupric sulfate?

3. What difference in observation would have occurred if you had used a solution of potassium chromate in studying the migration of ions?

4. Would a saturated solution of carbon dioxide in water have a low or a high conductivity?

5. Arrhenius assumes that the rate of migration of an ion is constant at a given temperature and under the influence of a given electric field. He also assumes that ionization increases on dilution. Can you suggest a possible way by which the molar conductance of a solution might increase upon dilution without further ionization?

CHAPTER XVIII

APPLICATION OF THE IONIC THEORY

87. Formation of a Precipitate. To each of the solutions (5 ml.) of sodium sulfate, saturated calcium sulfate, and dilute sulfuric acid, add a few drops of barium chloride solution. Note the character of the precipitate in each case. Is it the same? Should you expect a solution of any sulfate to give a precipitate with barium chloride? Why? Formulate the action in each of these instances in terms of ions. What is the molar solubility of barium sulfate? In like manner test the action of barium nitrate solution and barium hydroxide solution with dilute sulfuric acid. Would any soluble barium salt give a similar result? Why? Formulate the ionic action involved in each case.

88. Formation of a Gaseous Product. Add dilute sulfuric acid to a solution of sodium carbonate until no more gas is formed. Evaporate the solution to dryness. What is the residue? Does it contain any sodium carbonate? Make an ionic equation to indicate the reaction. Name other salts which are decomposed completely by dilute sulfuric acid; by concentrated sulfuric acid. State which is the more active in the ionic sense: normal sulfuric acid or normal hydrochloric acid. Why is sodium chloride completely decomposed by concentrated sulfuric acid but not by dilute sulfuric acid?

89. Formation of a Little Ionized Substance. Add a few drops of methyl orange to 10 ml. of water in a test tube. Add one or two drops of dilute hydrochloric acid to the tube. Note the change in color. Dissolve about 1 g. of sodium acetate in this solution and observe the changes in color. Make an ionic equation to show the action of hydrochloric acid on sodium acetate. Is acetic acid an active acid? Is it more active in the presence of an excess of sodium acetate or less active? Recall the action of sodium hydroxide and hydrochloric acid as illustrated in

Experiment 77. Perform a similar titration, using normal sodium hydroxide and white vinegar. Run the base from the burette into the acid. Use phenolphthalein as the indicator. Calculate the per cent of the acid in the vinegar. What is the normality of the acid?

90. Heat of Neutralization. (*Use the calorimeter described in Experiment 73.*) Prepare 150 ml. of 2-normal hydrochloric acid by diluting the concentrated acid (12-*N*) found in your reagent set. Obtain from the storeroom an equal (measured accurately) amount of 2-normal sodium hydroxide solution. Pour the hydroxide solution into the calorimeter beaker *A* and assemble the apparatus. Allow a few minutes for the solution to reach the temperature of the beaker; then read the temperature accurately. Raise the covers sufficiently to add the 2-normal acid to the solution in the calorimeter beaker. Stir the mixture, noting any change in temperature. Read the thermometer when the change is complete. Assume that the heat absorbed by the calorimeter is 12 calories. The specific heat of the solution may be assumed to be 1. The density may also be taken as being 1. Calculate the amount of heat lost or gained during this action. Heat of neutralization is defined as the amount of heat evolved when one gram equivalent of an acid reacts with one gram equivalent of a base in dilute solution. Calculate the heat of neutralization for hydrochloric acid and sodium hydroxide from the results of this experiment. What values should you expect had you used sulfuric acid (2-*N*) and potassium hydroxide (2-*N*)?

91. Use of Indicators to Measure Ion Concentrations. The colors given to solutions by indicators are often used as a measure of the concentration of hydrogen or of hydroxide ions. Using very dilute solutions of highly ionized (strong) acids (or bases), the shade of color due to various ion concentrations may be approximately established. By matching colors the ion concentrations of solutions of less highly ionized compounds may be approximated.

For example, if we select 0.01-*N* hydrochloric acid, the ionization is so nearly complete that little error will result if we assume it to be complete. On this assumption the solution is said to be 0.01-*N* with respect to hydrogen ions. If we now note the shade and depth of color given to this solution by certain indicators, we have a standard of comparison for other solutions of less highly ionized acids. Any acid solution which, using the same amount of solution and indicator, gives the same shade and depth of color is said to have a hydrogen ion concentration of 0.01-*N*.

For different ranges of hydrogen ion concentration different indicators are chosen. Each indicator is used in that range where it is known to be most effective.

a. Prepare 60 ml. of 1-*N* hydrochloric acid by adding distilled water to the proper volume of the laboratory reagent (12-*N*) and shaking the mixture. From this solution prepare between 50 and 100 ml. of 0.1-*N* acid, and using the latter make from 50 to 100 ml. of 0.01-*N* acid; also prepare a 0.001-*N* solution. On account of the error in measuring volumes by means of a graduated cylinder, and the probable variation of the concentration of the laboratory solution from 12-*N*, the concentrations of these solutions are known only approximately. What is the hydrogen ion concentration in each, assuming complete ionization?

Pour into marked test tubes 10 ml. each of 1-*N*, 0.1-*N*, 0.01-*N*, 0.001-*N* hydrochloric acid and pour 10 ml. of water into a fifth test tube. Add to each tube a single drop of methyl violet solution. Hold the tubes in a vertical position over a piece of paper, look down through the solutions, record the color of each solution, and note the smallest concentration of hydrochloric acid which shows with this indicator a color different from that with water. If the indicator solution is so dilute that one drop does not produce a distinct color, add from one to two more drops, but record the number of drops and use the same number in all the tubes. (The color in the more concentrated solution will fade on standing. It may be restored by adding another drop of the indicator.) Set aside the remainder of the solutions for use later.

b. Repeat the experiment with nitric acid *or* with sulfuric acid, using the same concentrations as before (*N*, 0.1-*N*, 0.01-*N*, 0.001-*N*). Compare the colors obtained with the different acids. Are the colors characteristic for each acid? If so, what substance determines the color? If 0.01-*N* hydrochloric acid is completely ionized, what conclusion can you draw with respect to the ionization of nitric acid and of sulfuric acid at the same concentration?

c. Methyl violet can be used to determine, approximately, concentrations of the hydrogen ion between N and 0.001-N. While the concentration of hydrogen ion in the 0.001-N solution (which is often written $10^{-3}\ N$) is small compared with that in the 1-N solution, it is ten thousand times as great as it is in pure water. Methyl orange may be used to examine solutions in which the concentration of hydrogen ion is between $10^{-3}\ N$ and that of pure water, $10^{-7}\ N$. By a tenfold dilution of the $10^{-3}\ N$ (0.001) HCl solution, prepare from 50 to 100 ml. of $10^{-4}\ N$ HCl, and from this prepare a $10^{-5}\ N$ solution. Test 10-ml. portions of these three solutions and of water with one drop of methyl orange as before. Save your solutions for later use.

d. Summarize your results with the two indicators in a table which shows the color obtained at various concentrations of H^+. Show your table to the instructor.

e. From the laboratory 6-N acetic acid prepare solutions which are approximately N, 0.1-N, and 0.01-N. Determine by means of indicators the approximate concentration of hydrogen ions in each of these solutions.* Place a 10-ml. portion of each of the solutions in a labeled test tube, place 10 ml. of distilled water in another test tube, and test each solution with methyl violet as in *b*. For comparison repeat the test with hydrochloric acid solutions of suitable concentrations. Estimate approximately the concentration of hydrogen ion in the N and 0.1-N solutions. Repeat the experiment, but use methyl orange instead of methyl violet, and estimate the concentration of H^+ in the 0.1-N and 0.01-N solutions. Calculate the fraction of the acetic acid which is ionized and the fraction which is un-ionized, using your data. The fraction of the acid which is in the form of ions is called the degree of ionization.

92. Displacement. Add 3 g. of mercury to 5 ml. of a silver nitrate solution in a 50-milliliter Erlenmeyer flask. Shake the contents of the flask vigorously for five minutes. If a white precipitate forms, add four drops of dilute nitric acid (6-*N*) to the flask and shake it until the precipitate dissolves. Decant the liquid into another flask of the same size. Wash the metallic residue in the first flask several times with water. Examine the residue carefully. What is it? To the solution in the second flask add 0.5 g. of copper wool. Shake its contents for a minute or two, and note the color of the solution and the appearance of the copper wool. Remove 1 ml. of this solution and treat it with a slight excess of ammonium hydroxide. What is your observation? Pour the remainder of the solution into a test tube and treat it with 0.1 g. of powdered zinc. Allow the tube to stand for a minute, and filter the solution. Test the filtrate by the addition of ammonium hydroxide. Is there any change in color?

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* In color comparisons it is not safe to trust the memory or even written descriptions.

What was the nature of the solution in the test tube before the final addition of ammonium hydroxide? Make ionic equations to show the successive displacement of the metals.

EXERCISES

1. What is meant by the primary and secondary ionizations of sulfuric acid?

2. Would methyl violet be a suitable indicator for the exact titration of sodium hydroxide with a sulfuric acid solution of known concentration?

3. Starting with a solution of potassium iodide, give ionic equations to show the changes which would occur if this solution and the resulting solutions were successively treated with bromine, chlorine, and fluorine.

4. Check the results obtained in Experiment 92 with the electromotive series of the metals.

CHAPTER XIX

SULFUR AND HYDROGEN SULFIDE

93. Physical Properties of Sulfur. *a.* Describe the physical properties of roll sulfur. Physical state? Color? Odor? Taste? Soluble in water? Is there any relation between the solubility of a substance in water and its having taste? What relation is there between the odor and volatility of a substance at room temperature?

b. Test the solubility of a small piece of roll sulfur in carbon disulfide. (Do not bring carbon disulfide near a flame!) Pour the mixture through a filter and collect the filtrate. Allow the solution to stand in a watch glass until the carbon disulfide has evaporated. Note the form of the sulfur crystals.

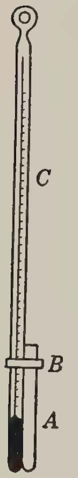

FIG. 46

c. Draw out a glass tube to form a rather large capillary. When cool, cut it in sections about 5 cm. long. Seal off the end of one of these sections to make a melting-point tube. Now pulverize in a mortar several of the crystals from *b*. Introduce some of this powdered sulfur into the melting-point tube (Fig. 46). Tap the tube until the sulfur reaches the bottom. Fasten the tube *A* to the lower end of a thermometer *C* with a rubber band *B*. The end containing the sulfur should be even with the bulb of the thermometer. Suspend the thermometer so that the bulb dips into some concentrated sulfuric acid in a small beaker. Do not allow the acid to touch the rubber band. Heat the acid slowly and stir constantly. Note and record the temperature at which the sulfur begins to melt.

d. Determine the density of roll sulfur by weighing accurately several small pieces and then immersing them in a graduated cylinder partly filled with distilled water. The volume of water displaced can be readily determined from the readings on the cylinder before and after immersing the sulfur. Assuming the weight of water to be exactly 1 g. per milliliter, we can calculate the density of sulfur as compared with the density of water.

94. Allotropic Forms of Sulfur. *a. Rhombic sulfur.* Recall the shape of the sulfur crystals obtained from the solution of roll sulfur in carbon disulfide. These were crystals of the most usual allotropic modification of sulfur.

b. Monoclinic sulfur. Fill a small crucible about two thirds full of roll sulfur. Heat it carefully until all the sulfur is melted. Allow it to cool, and when a crust has just covered the surface, make a hole in it with the sharp end of your deflagrating spoon and pour out the liquid interior. Note the shape of the sulfur crystals remaining in the crucible. Test their solubility in carbon disulfide. What form of sulfur will be obtained by allowing the carbon disulfide to evaporate?

c. Sulfur λ and sulfur μ. Place 10 g. of roll sulfur in a test tube and heat it very slowly, watching the changes in color and state. When the sulfur is first melted, will it pour out? Is it a thin liquid or a thick one? What is the case at a somewhat higher temperature (180°)? at a much higher temperature? Continue heating until the sulfur boils; then pour it quickly into a dish of cold water. Describe the product. Test its solubility in carbon disulfide. Preserve the material.

Heat another portion of sulfur until it is just melted. Pour it into water. Examine this product and determine whether or not it will dissolve in carbon disulfide. How can you explain these results? Write the equation. After about a week, again examine these products and find out whether they will dissolve in carbon disulfide. Explain any changes. What is the transition point of sulfur λ into sulfur μ?

95. Chemical Properties of Sulfur. *a.* Study the action of metals with sulfur by dropping small pieces of copper, iron, and tin into separate test tubes of hot sulfur. Write equations for the chemical actions concerned.

b. Boil a small amount of powdered sulfur with a few milliliters of sodium hydroxide solution

$$(6\ NaOH + 4\ S \longrightarrow 3\ H_2O + Na_2S_2O_3 + 2\ Na_2S)$$

Allow the undissolved sulfur to settle, pour off the supernatant liquid, and add to it a few drops of a solution of sodium nitroprusside ($Na_2Fe(CN)_5(NO)$). Note the reddish-violet color. The color is produced by a compound having a probable formula $Na_3Fe(ONSNa)(CN)_5$.

96. Preparation of Hydrogen Sulfide. *a.* Try the action of dilute hydrochloric acid on separate portions of several solid sulfides (***CARE, HOOD!***) and test any evolved gas with filter paper moistened with a solution of lead acetate. Try ferrous sulfide, lead sulfide, and zinc sulfide. Is hydrogen sulfide given off in each case? Write equations for the chemical actions noted.

b. Fit up a 250-milliliter Erlenmeyer flask to serve as a generator for hydrogen sulfide. Place in it 5 g. of ferrous sulfide and add 20 ml. of dilute hydrochloric acid. Collect a bottle of hydrogen sulfide by displacement of air. Plunge a lighted splint into it. What is the result? Write the equation for the burning of hydrogen sulfide.

97. Properties of Hydrogen Sulfide. *a.* Saturate 2 ml. of water with hydrogen sulfide. Use a pressure flask when treating any solution with hydrogen sulfide. How can you tell when the solution is saturated? Test the reaction of the liquid with litmus.

b. Boil the solution from *a.* Can the gas be completely removed by this process? Repeat, using dilute hydrochloric acid. Is all the HCl removed by boiling? How can you tell? If you note any differences in the behavior of the two solutions, explain them.

c. Carefully test the action of hydrogen sulfide water on brightly polished strips of iron. Is there any action? Repeat, using dilute sulfuric acid. Explain any differences in activity in terms of the ionic theory.

d. Pass hydrogen sulfide into a mixture consisting of 1 ml. of potassium dichromate solution and 5 ml. of dilute sulfuric acid. Observe the changes that occur. When the action is complete, filter the solution. Of what does the solution consist? What is the product on the filter? Write the equation.

98. Sulfides. *a.* Place in each of a series of test tubes 1 ml., respectively, of solutions of mercuric chloride, copper sulfate, cadmium sulfate, zinc sulfate, and ferrous sulfate. Dilute each to 10 ml. and saturate with hydrogen sulfide. Give the colors of the precipitates and write equations to show the reactions that take place. Thoroughly shake the first test tube and quickly divide the contents into approximately equal portions. Add 5 ml. of dilute hydrochloric acid to one portion and 5 ml. of concentrated hydrochloric acid to the other portion. Repeat this process with each of the other test tubes. Record your observations. From these experiments suggest a way of separating zinc from copper.

b. Dilute 1 ml. of a solution of ferrous sulfate to 10 ml. and add to it 1 or 2 ml. of ammonium sulfide. Is the precipitation more or less complete than was the case when the solution of the sulfate was saturated with hydrogen sulfide? Explain in terms of the ionic theory.

c. Saturate 5 ml. of a solution of sodium hydroxide with hydrogen sulfide. What is the product? Give equations. To the solution add 5 ml. more of the sodium hydroxide. What is the product? Give the equation. To this solution add a little powdered sulfur. What compound does sulfur form with the previous product?

EXERCISES

1. Recall the action of sulfur with oxygen. What was the compound formed? What was its odor?

2. What is the transition point of rhombic and monoclinic sulfur? Is the change from the first into the second, at this temperature, accompanied by the absorption or evolution of heat? Upon what do you base your conclusion?

3. What are the possible products obtained by burning hydrogen sulfide in air?

4. Is hydrogen sulfide gas stable at ordinary temperature?

5. Should you expect silverware to tarnish more rapidly in air containing hydrogen sulfide or in air containing hydrogen sulfide and sulfur dioxide? Give reasons for your answer.

CHAPTER XX

OXIDES AND OXYGEN ACIDS OF SULFUR

99. Sulfur Dioxide. *a.* Burn a piece of sulfur in a deflagrating spoon. Note the odor. What is the color of the flame? What is the product of the burning? Write the equation.

b. Heat strongly some powdered iron pyrites in a hard-glass test tube. Is any sublimate formed? Try the action of hydrochloric acid on the residue and also on some unheated pyrites. Is there any difference in the behavior of the heated pyrites and the original pyrites? During these tests what substances could you recognize by their odor, by their color, or by other properties? Express the changes by means of equations.

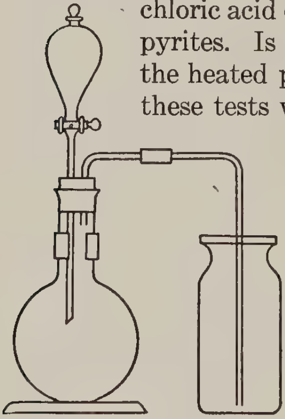

FIG. 47

c. Just cover a little powdered charcoal in a test tube with concentrated sulfuric acid. Heat the mixture cautiously and note the odor of the evolved gas. Write an equation for the action. What is the function of the carbon?

d. Repeat Experiment *c*, using separately small pieces of copper and sulfur. What is the gas given off? What is the function of the copper? of the sulfur? Write the equations.

100. Preparation of Sulfur Dioxide. Sulfur dioxide may be prepared in the laboratory by the action of dilute sulfuric acid on sodium hydrogen sulfite or on sodium sulfite. When cylinders of liquid sulfur dioxide are available, the following preparation may be omitted.

Set up a Florence flask provided with a dropping funnel (Fig. 47), or thistle tube if funnel is not available, and a delivery tube. Place 12 g. of sodium hydrogen sulfite in the flask. Test the apparatus to see that it is air-tight. Then allow dilute sulfuric acid from the dropping funnel to fall on the salt. Fill four wide-mouthed bottles with the gas by upward displacement of air.

101. Properties of Sulfur Dioxide. *a.* Place in one of the bottles a moistened strip of colored calico. Is it bleached? Some calicoes are bleached by sulfur dioxide; others are not. Try a different calico. What is the result?

b. Test the action of the oxide on a strip of moistened blue litmus paper when it is placed in the second bottle. What color changes are seen? Explain.

c. To the third bottle add 5 ml. of water. What is this solution called? Allow time for the gas to dissolve. Pour the solution into a test tube and add barium chloride solution. What is the precipitate? Is it soluble in hydrochloric acid? Write the equations.

d. Dissolve the gas in the last bottle in water as before. Then add to the solution a few drops of potassium permanganate solution. Give the result and write the equation.

102. Sulfur Trioxide. *a.* What would happen if sulfuric acid were heated to its boiling-point?

b. What is *fuming sulfuric acid*? What is *oleum*?

c. Sulfur trioxide exists in two forms. What word is used to express this fact?

d. When concentrated sulfuric acid is boiled, why is a dense white fog produced?

e. State which is the more stable toward heat: sulfur dioxide or sulfur trioxide.

f. How may sulfur trioxide be made from sulfur dioxide?

103. Preparation of Sulfuric Acid. Arrange an apparatus for making sulfuric acid as shown in Fig. 48. Study carefully the use of each part. Generator *A* is to be used for preparing sulfur dioxide. The small flask *B* should have a one-hole stopper and contain about 10 ml. of concentrated nitric acid. The 1000-milliliter Florence flask *C* is fitted with a four-hole rubber stopper. Pass a dropping funnel through one hole. Connect the flask with the generator *A* and the small flask *B* by right-angled delivery tubes and short pieces of rubber tubing. The delivery tubes should come nearly to the bottom of flask *C*. The fourth hole carries a delivery tube reaching just through the stopper. Connect this with a wash bottle *D*, partly filled with water, and connect the wash bottle with the vacuum line (an aspirator will serve as a substitute).

Make sure that all connections are tight and that the purpose and method of operation of the experiment are clearly understood. Then start drawing a very slow stream of air through the apparatus. Begin the generation of sulfur dioxide. Warm the flask containing the nitric acid.

What action is taking place in the generator *A*? What gases are being supplied from the nitric acid? Shut off the supply of gases from the nitric-acid flask. When white crystals form on the flask *C*, introduce about 10 ml. of water from the dropping funnel. Why do the crystals disappear? Why do brown fumes reappear in the flask? Preserve the sulfuric acid for the following experiment. A sulfur-dioxide tank may be used instead of a generator.

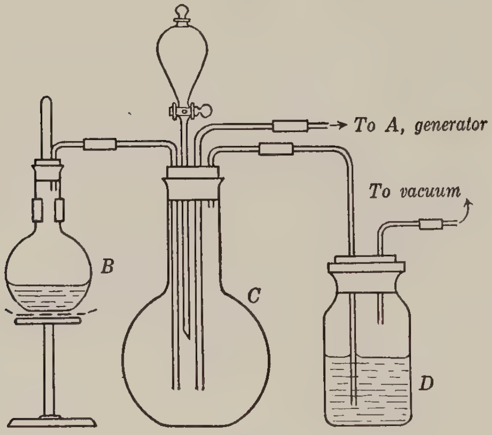

FIG. 48

104. Properties of Sulfuric Acid. *a.* Test the action of dilute sulfuric acid on litmus, on sodium carbonate, and on metallic zinc. Write the equations. What class of compounds give these reactions?

b. Pour a few drops of concentrated sulfuric acid on a little sugar in a test tube. Explain the action which takes place. Place a crystal of copper sulfate in a test tube and cover it with concentrated sulfuric acid. Allow the tube to stand for at least an hour. Do you see any change in color? What property of sulfuric acid is illustrated?

c. Recall the action of concentrated sulfuric acid on carbon and on copper. What property of sulfuric acid was illustrated? Write the equations.

d. Add to 2 ml. of sulfuric acid a few drops of barium chloride. Write the equation. Is the precipitate soluble in hydrochloric acid? How can sulfites be distinguished from sulfates?

105. Sulfates. *a.* Mix 0.1 g. of sodium sulfate with 0.5 g. of sodium carbonate and place the mixture in a cavity on a piece of charcoal. Ignite the mixture before the blowpipe, and place some of the fused mass on a silver coin. Moisten the mass with dilute hydrochloric acid, and note the darkening of the coin and the odor of the gas evolved. This procedure is sometimes called the Hepar test. Write all equations for the chemical actions concerned.

b. (*Optional*) Devise a method (Reference) for making barium nitrate or strontium nitrate from the corresponding sulfate. Submit your plan to your instructor before starting work on the preparation.

EXERCISES

1. State some industrial uses of sulfur dioxide.

2. Why do we believe that there is such a compound as sulfurous acid?

3. List the advantages of the contact process for sulfuric acid over the chamber process.

4. What causes "sulfuric-acid mist" when the concentration of sulfuric acid used in absorbing sulfur trioxide becomes too low?

CHAPTER XXI

THE INERT GASES

106. Preparation of Argon. (*Optional*) The compressed oxygen of commerce contains about 3 per cent of argon. It also contains smaller amounts of other inert gases and some nitrogen.

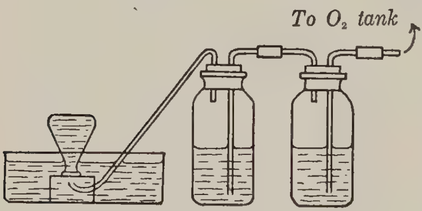

FIG. 49

Connect an oxygen tank with two absorption bottles as shown in Fig. 49. Make a pyrogallol solution by dissolving 25 g. of pyrogallol in 100 ml. of water. Pour half of this solution into each bottle. Add an equal volume of sodium hydroxide solution to each bottle. Stopper the bottles immediately. Allow the delivery tube from the second bottle to dip under the mouth of a 50-milliliter flask filled with water and inverted in a pneumatic trough. Turn on the oxygen and regulate its flow so that *only one* bubble of gas enters the flask every five seconds. When the flask is filled with the gas, shut off the oxygen supply. Introduce a burning splint into the flask. What happens? Explain. In what way could the nitrogen be removed from the argon and other inert gases? How could argon be separated from helium.

EXERCISES

1. Why are the inert gases so named?

2. In what way did helium receive its name?

3. What is the source of commercial helium?

4. If a balloon filled with hydrogen will lift 6 tons, what weight would the same balloon filled with helium lift?

5. How did neon get its name?

6. To what use has neon been put?

7. What is the relative rate of diffusion of neon and helium?

8. What is the partial pressure of neon in the air, atmospheric pressure being 760 mm.?

9. A room is 20 m. long, 10 m. wide, and 4 m. high. At a temperature of 20° and a pressure of 740 mm., what weight of argon is present? what weight of helium?

10. In what country has helium been obtained in large amounts?

11. What is the source of radon? State one important use of the gas.

12. For what purpose is argon mainly used?

CHAPTER XXII

THE PERIODIC SYSTEM AND THE STRUCTURE OF THE ATOM

107. Building Atomic Models. (*Two students may work together.*) The instructor will assign to each pair of students the model of an element to construct. Consult the text, if necessary, and have in mind the arrangement of electrons in the assigned element. Each pair of students will need pliers.

Obtain from the storeroom some 16-gauge galvanized iron wire and a solid rubber ball (half an inch in diameter) to represent the nucleus. Students constructing the models of the lithium atom will need, in addition, three rubber balls of a color different from that of the ball which is to represent the nucleus. The model of each succeeding element requires one more of these balls than the preceding one; for example, beryllium requires four; boron, five; carbon, six; and so on (Fig. 50).

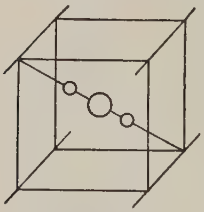

FIG. 50

Construct a 4-inch cube of the wire. Solder the wires together, using rosin-centered solder and the Bunsen flame. Allow a wire to project 1 in. beyond each corner of the cube. Connect a wire bearing the balls representing the nucleus and the first two electrons to diagonally opposite corners of the cube, as shown in the figure. Place balls at the corners of the cube until the desired model is complete. (Lithium would need but one ball; neon would need eight.)

If the assigned element is one found in the third period of the periodic table, a second cube larger than the first must be made. A second diagonal wire will be necessary to hold it in place. Other arrangements than those described should suggest themselves to the students interested in building more complex models.

Using the symbols *P* and *E* for protons and electrons, respectively, print on the ball representing the nucleus the number of protons and electrons in the nucleus of the element represented.

EXERCISES

1. Which should you expect to be the more acidic: Cr_2O_3 or CrO_3?

2. In what group should you expect to find an element which exhibits a maximum valence of 8? of 7? Does fluorine ever have a valence of 7?

3. What classes of compounds are formed by the nonmetallic elements?

4. Is the specific heat of the elements a periodic function?

5. Which element has the greatest atomic volume?

6. Does the arrangement of the electrons in the outer shells of the atoms explain in any way the periodic law? Compare the arrangement in the outer shell of elements of the same family.

7. In the light of present information criticize the older statement of the periodic law.

CHAPTER XXIII

NITROGEN AND ITS HYDRIDES

108. Preparation of Nitrogen. *a.* Secure a piece of white phosphorus (*CARE!*) from the storeroom and keep it under water until you are ready to use it. Why? Cover the bottom of a pneumatic trough with water to the depth of 3 cm. Float upon the water a crucible carrying the phosphorus. Ignite the phosphorus by touching it with the hot end of a deflagrating spoon. Quickly lower a beaker over the crucible, being sure that the rim of the beaker is kept below the surface of the water. Note that the water rises in the beaker. Explain. Allow the apparatus to remain until the fog of phosphorus pentoxide has disappeared. Add water to the trough. With your fingers, tip over the crucible so that it fills with water and sinks. Adjust the beaker so that the liquid stands at the same level within and without the beaker. Make a mark at the water level. Close the beaker with a glass plate and set it upright. Remove the plate and introduce a burning splint into the gas. What is the result? Measure the water needed to fill the beaker to the mark. Also find the total volume of the beaker. From these values calculate the decrease in volume of the air.

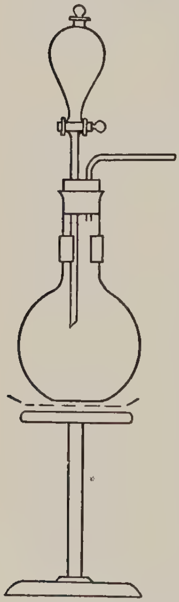

FIG. 51

b. To 4 g. of ammonium chloride and 5 g. of sodium nitrite, add 25 ml. of water and thoroughly shake the mixture. Place these materials in a 250-milliliter flask fitted with a delivery tube and a dropping funnel (Fig. 51). Heat the flask gently but constantly with a low flame in order to obtain a continuous flow of nitrogen.

Collect two bottles of the gas by the downward displacement of water. Give an equation for the reaction.

NOTE. If the action becomes too vigorous, remove the flame and allow water to run into the flask from the funnel. Be sure to remove the delivery tube from the trough as soon as the last bottle is filled. Why?

109. Properties of Nitrogen. *a.* Test the first bottle of nitrogen prepared in Experiment 108, *b*, with a glowing splint and then with a lighted splint. Is the gas a supporter of combustion?

b. By means of a deflagrating spoon introduce burning sulfur into the second bottle. What is the result? Explain. Remove the spoon. Is the sulfur still burning?

110. Preparation of Ammonia. *a.* Heat 1 ml. of ammonium hydroxide. Cautiously observe the odor of the gas given off. What is it?

b. Add 2 ml. of dilute sodium hydroxide solution to five or six crystals of ammonium sulfate. Heat the mixture gently and again observe the odor of the escaping gas. What is it? Write the equation. Would other ammonium salts act in a similar way?

c. Cover the bottom of a test tube with powdered gelatin. Add to this 0.5 g. of powdered soda lime. Heat the tube in the Bunsen flame. Note the odor of the gas. What is it? Would all organic substances give similar results? Name several substances which might be used instead of gelatin.

d. Mix intimately 10 g. of ammonium chloride and 10 g. of calcium oxide. Place them in a 250-milliliter flask fitted with a two-hole rubber stopper carrying a dropping funnel and a delivery tube. Add 5 ml. of water through the funnel. Heat the flask gently, if necessary, to cause the action to start. Collect three bottles of the gas by downward displacement of air. Cover each with a glass plate.

111. Properties of Ammonia. *a.* Test the gas in one bottle from Experiment 110, *d*, with a lighted splint and with a glowing splint. What is your conclusion?

b. Place the mouth of the second bottle under water in a pneumatic trough and then remove the glass plate. Does the water enter the bottle? Why? Remove the bottle, and test the water in it with a piece of red litmus paper.

c. Pour 1 ml. of concentrated hydrochloric acid into a dry bottle. Rotate the bottle until the acid has touched all parts, shake out any excess acid, and invert it upon the third bottle of ammonia. Remove the glass plate and hold the bottles together firmly. Explain the observed action. Write an equation for the action.

EXERCISES

1. What weight of nitrogen may be obtained by heating together the amounts of ammonium chloride and sodium nitrite given in Experiment 108, *b*? Which salt is present in excess? By how much? What would be the volume of this nitrogen at standard temperature and pressure? at 30° and 730 mm.?

2. What weight of magnesium nitride will be formed by heating 5 g. of magnesium in a current of nitrogen?

3. What weight of ammonia can be formed by decomposing 15 g. of magnesium nitride with excess water?

4. If 10 g. of ammonia are dissolved in 500 ml. of water, how many milliliters of 1-*N* hydrochloric acid will be needed to neutralize the base so formed?

CHAPTER XXIV

OXIDES AND OXYGEN ACIDS OF NITROGEN

112. Nitrous Oxide. Place 15 g. of ammonium nitrate in a 100-milliliter pyrex flask. Fit it with a one-hole rubber stopper bearing a delivery tube dipping into water in a pneumatic trough (Fig. 52). Heat the flask gently, and after the air has been driven out collect two bottles of nitrous oxide by downward displacement of water. Write the equation showing the decomposition of ammonium nitrate. Record the observed physical properties of nitrous oxide. Test the gas in the first bottle with a glowing splint. What is the result? Test the gas in the second bottle with burning sulfur. The spoon containing the sulfur should be hot. What is the result? Write equations for the chemical actions observed. To what use is nitrous oxide often put? By what other name is it sometimes known?

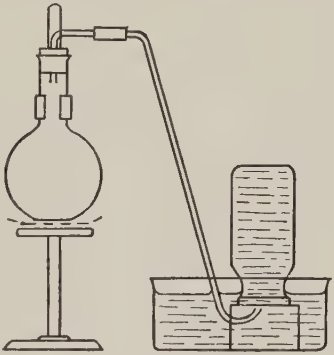
FIG. 52

113. Nitric Oxide. Use the same apparatus as in the preceding experiment, except that a thistle tube is added. Place in the flask 10 g. of copper turnings. Add 10 ml. of water and adjust the thistle tube so that its lower end comes below the water level. Add a few milliliters of concentrated nitric acid through the funnel as needed. Write the equation for the chemical action. Collect two bottles of the gas by displacement of water. Note its physical properties. Test the gas in the first bottle with a glowing splint. What result is observed? Admit a bubble of air to the second bottle, keeping it inverted over water in the pneumatic trough. What causes the color observed? Does this color persist? Explain.

114. Nitrogen Dioxide. Heat 0.5 g. of lead nitrate in a test tube. Note the color and odor of the gas evolved. Write the equation for the action.

115. Preparation of Nitric Acid. Place 10 g. of sodium nitrate in a retort and add 5 ml. of concentrated sulfuric acid. Arrange the retort so that its neck reaches well inside the mouth of a large test tube resting in a beaker of ice water (Fig. 36). Heat the retort carefully and note the color of the distillate. What causes the brown fumes? (Take great care to keep the acid from touching the clothes or the skin.) Write the equation for the preparation of nitric acid by this method.

116. Properties of Nitric Acid. *a.* Dilute a little concentrated nitric acid with three times its volume of water, pour the dilute acid upon a few pieces of copper in a test tube, and heat the tube. What is the color in the test tube as the action begins? Hold a piece of white paper behind the test tube for a background and compare the color of the gas at the mouth of the tube with that inside the tube. Account for all facts observed. Write equations for the chemical changes involved.

b. Using a test tube, pass hydrogen sulfide into about 3 ml. of dilute nitric acid. What do you observe? What causes the murky appearance. Test the solution for a sulfate. What is the result? Write the equation for the action.

c. Add some dilute nitric acid to a few pieces of iron. Is a colored gas evolved? Test the solution for ammonium compounds by adding sodium hydroxide until the solution is strongly alkaline to litmus. Warm the mixture. Can you detect the odor of ammonia? Test the vapor with moist litmus. Explain all actions.

d. Add a piece of iron to a solution of copper sulfate. Immerse a second piece of the iron in fuming nitric acid (*CARE!*). Does the iron react? Pour off the acid, rinse the iron with distilled water, and quickly place it in copper sulfate. Remove it from the copper sulfate solution, heat to redness, and again place it in the solution. What results are observed?

e. Add about 3 ml. of concentrated nitric acid to some aluminum. Is the action as vigorous as you would expect from the position of aluminum in the electromotive series and the activity of concentrated nitric acid as an oxidizing agent? The reduction of the activity of iron by means of oxidizing substances, such as zinc chromate, basic lead chromate, and red lead (Pb_3O_4), is made use of in anti-corrosion paints.

117. A Test for Nitrates. Prepare a saturated solution of ferrous sulfate or ferrous ammonium sulfate and add a few drops of a solution made by placing a crystal of sodium nitrate in 5 ml. of water, taking care that the two liquids are well mixed. Place 5 ml. of concentrated sulfuric acid in a test tube and pour the ferrous sulfate solution carefully down the side of the inclined tube. The sulfate solution will float on the acid. The brown ring which appears at the juncture of the liquids is a characteristic test for nitrates, provided no iodides, bromides, or nitrites are present. The formula for the compound which colors the ring brown is $FeNOSO_4$. Write equations to show all the reactions.

118. Preparation of Sodium Nitrite. *a.* Heat in an iron dish a mixture of 5 g. of sodium nitrate and 10 g. of metallic lead. When the mass has become dark brown, cool and extract it with hot water. Test a small portion of the clear extract with a few drops of sulfuric acid. Explain the observed phenomena. Test another small portion with a solution of potassium iodide. Add a drop of dilute acid. Explain. Write equations for all the actions.

b. Repeat Experiment 117, using 5 ml. of the extract from *a* instead of the nitrate solution, and acetic acid instead of sulfuric acid. This is a test for nitrites. Write all equations.

119. Reduction of Nitrates to Ammonia. Dissolve 0.5 g. of sodium nitrate in 3 ml. of water in a test tube. Add 3 ml. of concentrated sodium hydroxide solution and a few pieces of metallic aluminum. Heat the mixture and test the escaping gas with litmus. What is the result? Write the equation showing the reduction.

EXERCISES

1. How many liters of nitrous oxide would 100 g. of ammonium nitrate yield?

2. How many liters of nitric oxide can be obtained from 20 g. of copper?

3. What weight of nitric acid will be formed when an excess of sulfuric acid acts on 200 lb. of sodium nitrate?

4. Write equations showing the chemical changes involved in the synthesis of nitric acid from the nitrogen of the air.

5. Write equations showing the chemical changes involved in the formation of nitric acid by the oxidation of ammonia.

6. What are the products formed by heating ammonium nitrite? Write the equation.

7. Would nitrites be changed to ammonia by the method used in Experiment 119?

8. List the possible reduction products of nitric acid.

9. Show clearly how the oxides of nitrogen illustrate the law of multiple proportion.

CHAPTER XXV

OXIDATION AND REDUCTION

NOTE. Balance all equations for reactions involving oxidation and reduction by making use of the transference of electrons.

120. Formation of Compound Ions. *a.* Treat 0.1 g. of powdered sulfur in an evaporating dish with 10 ml. of concentrated nitric acid. By applying heat, evaporate all but about 2 ml. of the acid (***HOOD!***). Dilute the resulting solution to 10 ml., filter it, and add a few drops of barium nitrate solution to the filtrate. What ion is shown to be present by this test? Write equations.

b. Dilute 1 ml. of a solution of potassium iodide to 10 ml. and pass chlorine into the liquid until all the iodine, which is liberated, disappears (***HOOD!***). To half the solution add four or five drops of silver nitrate solution. Is the precipitate soluble in ammonium hydroxide? What is the precipitate? Pass sulfur dioxide into the other half of the solution. Expel the dioxide by boiling the solution and then treat the liquid with silver nitrate solution. Is the precipitate soluble in ammonium hydroxide? What is it? Write equations, except those with ammonium hydroxide.

What is formed when iodine is heated with concentrated nitric acid? Which substance is the oxidizing agent? Name a chemical change in which bromine atoms lose electrons.

121. Destruction of Compound Ions. *a.* Dissolve a crystal of potassium chlorate in 1 ml. of water. Treat the solution with an equal volume of concentrated hydrochloric acid. Warm the solution, and note the color and odor of the evolved gas. What is it? Which is the most vigorous oxidizing agent: Cl_2°, or Cl^-, or Cl^+? In what way can you account for the oxidizing activity of sodium hypochlorite?

b. Pass hydrogen sulfide into 1 ml. of concentrated nitric acid. What is the solid matter produced?

EXERCISES

1. Tell which of the following involve oxidation and reduction:

a. Action of sulfur trioxide on sulfuric acid.
b. Union of copper with chlorine.
c. Change of a nitrate into a nitrite.
d. Formation of Cl_2 from 2 Cl^-.
e. Displacement of H_2 from H_2SO_4.
f. Union of Cl^- with Ag^+ to form AgCl.
g. Union of CaO with SO_3 to form $CaSO_4$.

2. Give the valence of the following elements when they are completely oxidized and completely reduced: nitrogen, chlorine, sulfur, fluorine, hydrogen, bromine, and selenium.

3. Balance the following equations and indicate the transference of electrons:

a. $HBrO_3 + HBr \longrightarrow Br_2 + H_2O$.
b. $HBrO_3 + H_2SO_3 \longrightarrow H_2SO_4 + HBr$.
c. $H_2S + H_2SO_4 \longrightarrow H_2O + S + SO_2$.
d. $HNO_3 + Zn \longrightarrow NH_4NO_3 + Zn(NO_3)_2 + H_2O$.
e. $Al + HCl \longrightarrow AlCl_3 + H_2$.

CHAPTER XXVI

PHOSPHORUS, ARSENIC, ANTIMONY, AND BISMUTH

122. Properties of Phosphorus. *a.* Recall the actions of oxygen and chlorine on phosphorus. What do these illustrate with respect to the activity of phosphorus toward such elements? Compare this behavior with that of nitrogen.

b. Boil in a test tube 0.5 g. of red phosphorus with 5 ml. of dilute nitric acid. Evaporate the solution to a sirup on the water bath. Add a few drops of concentrated nitric acid, and evaporate again. Repeat but do not evaporate the solution quite to dryness. What is the product?

c. Drop into an evaporating dish containing from 10 to 15 ml. of water one small piece of calcium phosphide. Test the gas with moist litmus papers. Compare the result with that obtained with ammonia.

123. Phosphoric Acids. *a.* Into 45 ml. of cold water sprinkle gradually about 2 g. of phosphorus pentoxide and allow the solution to stand until clear. What product is formed? Use the solution in *b, c, d,* and *g.*

b. To 3 ml. of the solution from *a* add a few drops of barium nitrate solution. What is the result?

c. Repeat *b,* using silver nitrate solution.

d. Pour the remainder of the solution, except that reserved for *g,* into a small flask and add two drops of nitric acid. Equip the flask with a one-hole stopper into which has been inserted a long piece of glass tubing. Boil the solution gently for about half an hour. (The tube will act as a condenser.) Remove the stopper, and continue heating for five minutes to expel the nitric acid. Cool the solution, dilute it to 15 ml. with water, and use it in *e, f,* and *g.* What is the purpose of the nitric acid?

e. Test portions of the solution from *d* with barium nitrate and with silver nitrate. What result is observed? Compare these results with those obtained in *b* and *c.*

f. To another part of the solution *d* add ammonium hydroxide until the solution is alkaline, then add magnesia mixture. The precipitate is $MgNH_4PO_4$.

g. Add two drops of the solution to 5 ml. of ammonium molybdate solution (in nitric acid) and heat the liquid to 90°, but do not boil it. What is the result? Repeat this test, using the solution from *a*. Is the result the same?

124. Phosphates. *a.* Test solutions of primary, secondary, and tertiary sodium phosphates with litmus papers. Account for the results. Place 5 ml. of a solution of tertiary sodium phosphate in each of two test tubes and add to one dilute nitric acid, drop by drop, until the solution is neutral. Now add to each a few drops of silver nitrate solution. Account for the difference observed.

b. Heat strongly over a blast lamp for about twenty minutes 1 g. of secondary sodium phosphate. After the substance has cooled, dissolve it in water and add dilute nitric acid, drop by drop, until the solution is just acid. Test the solution with silver nitrate and with barium nitrate. What are the results?

c. Heat, as in *b*, 1 g. of microcosmic salt ($NaNH_4HPO_4$). Test the vapor with moist litmus papers. Cool the residue and dissolve it in water. Acidify as in *b*, and test the solution with silver nitrate and with barium nitrate. What is the result? How would you distinguish between orthophosphate, pyrophosphate, and metaphosphate ions?

d. Make a small loop in a platinum or nichrome wire, heat it in a flame, and collect a bead of microcosmic salt. Heat the bead and touch it to a minute fragment of copper oxide. Heat it again and observe the color of the bead after it cools.

125. Chlorides of Phosphorus. *a.* Recall the action of water on phosphorus tribromide and on the triiodide. Obtain 2 ml. of phosphorus trichloride in a test tube. Breathe across the mouth of the tube. Explain your observation. Add to the chloride, drop by drop, about 5 ml. of water and boil the mixture. Test the vapor with litmus papers and with a glass rod moistened with ammonium hydroxide. What results are observed? Is this reaction reversible? Evaporate the residue to a small bulk, cool, dilute it with water, and divide the solution into two parts.

b. To one part of the solution from *a* add a few drops of silver nitrate, and to the other a few drops of copper sulfate. Heat each to boiling. What are the results? What property of phosphorous acid is hereby illustrated?

126. Properties of Arsenic. *a*. Examine a particle of arsenic. What class of elements does it resemble in appearance? Heat it in a hard-glass tube and cautiously note the odor.

b. Grind in a mortar 0.5 g. of arsenic trioxide with powdered charcoal. Heat strongly in a hard-glass tube and note the product. What is the action of the charcoal?

127. Tests for Arsenic. (*Optional*) *a*. The Gutzeit test for arsenic (*HOOD!*) may be carried out as follows: Place a few granules of pure zinc in a test tube (Fig. 53), cover them with 5 ml. of dilute sulfuric acid, and add from two to three drops of sodium arsenite solution to the mixture. Push a wad of cotton a third of the way down the tube and, supporting the latter in a vertical position, place over its mouth a piece of filter paper. Now place on the filter paper a crystal of silver nitrate and carefully observe any changes in the latter.

b. The Marsh test for arsenic (*HOOD!*) may be used instead of the Gutzeit test. Arrange the apparatus for this test as illustrated in Fig. 54. The tube *B* contains granular calcium chloride with plugs of glass wool at either end. The rubber hose connecting the calcium chloride tube with the generating flask should be previously boiled with sodium hydroxide solution. The tube *C* is of hard glass and is drawn out in the manner illustrated.

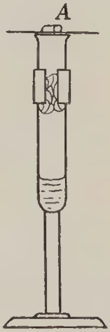

FIG. 53

Place 4 g. of zinc in the generating flask *A*, and pour water through the funnel until the end of the stem is just immersed; then add 25 ml. of dilute sulfuric acid (1 part of concentrated acid to 3 parts of water). As soon as the air has been completely displaced from the generator (*TEST, CAUTION!*), light the gas at the nozzle *D*. Hold a porcelain dish in the flame, and note whether or not a deposit is formed. If no deposit is formed, the reagents are free from arsenic. Add, through the funnel, from two to three drops of sodium arsenite solution and wash it down with a few drops of sulfuric acid. Note any change in the color of the flame. Hold a porcelain dish in the flame, and note the appearance of the deposit. Now heat the tube *C* strongly at the constriction. The appearance of a silvery mirror in front of or in the constriction is due to the deposition of free arsenic. Fill the flask *A* with water to stop further action. Treat the deposit on the porcelain dish with a few drops of sodium hypochlorite solution. What result is observed?

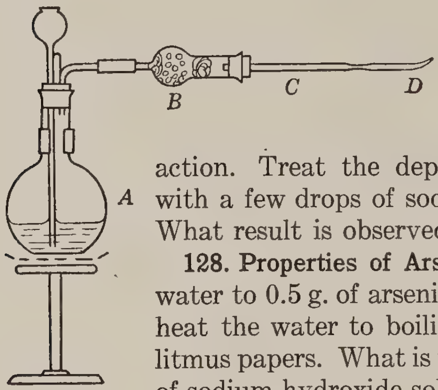

FIG. 54

128. Properties of Arsenious Compounds. *a.* Add water to 0.5 g. of arsenious oxide in a test tube and heat the water to boiling. Test the solution with litmus papers. What is the result? Add a few drops of sodium hydroxide solution and boil the mixture. Explain the result.

b. Boil 0.5 g. of arsenious oxide with concentrated hydrochloric acid. Is this action reversible? To answer this question, allow the solution to cool, and examine the crystals. Explain.

c. Consider carefully the results obtained in *a* and *b* and also those in Experiment 126, *a*, and then state which class of elements arsenic resembles.

d. Saturate with hydrogen sulfide a solution of arsenious oxide in hydrochloric acid. What do you observe? Remove and wash the precipitate. Pour over the precipitate ammonium sulfide and collect the filtrate in a fresh beaker. What is the result? Add dilute hydrochloric acid to the filtrate. What is the result? Explain.

129. Properties of Arsenic Compounds. *a.* Add two drops of sodium arsenate solution to 5 ml. of a solution of ammonium molybdate, and warm the mixture. What other ions have you encountered that behave similarly in the presence of ammonium molybdate?

b. Add magnesia mixture to 5 ml. of sodium arsenate solution. What other ion is precipitated by this reagent?

130. Properties of Antimony. (*Optional*) *a.* Repeat Experiment 126, *b*, substituting antimony trioxide. What are the results?

b. Boil 2 g. of powdered antimony with 10 ml. of concentrated nitric acid (***HOOD!***), dilute the acid with water, filter it, and wash the precipitate with a little sodium carbonate solution and then thoroughly with water. What is the product?

131. Properties of Antimonious Compounds. *a.* Boil antimony trioxide with water. Test the solution with litmus papers. What result is observed? Add sodium hydroxide to the oxide. Explain the result.

b. Boil another portion of the oxide with dilute hydrochloric acid. Explain.

c. Add two or three drops of antimonious chloride to 10 ml. of water. Devise a means of showing whether or not this reaction is reversible, and try it. What is the result?

d. Add a few drops of antimonious chloride to 5 ml. of dilute hydrochloric acid, and saturate the solution with hydrogen sulfide. What is the result? Filter, and treat the precipitate with ammonium sulfide. What is the result? Add dilute hydrochloric acid to the filtrate. Explain.

132. Properties of Bismuth Compounds. *a.* Grind a crystal of bismuth nitrate, add a little sodium carbonate, and heat the mixture on charcoal in the reducing flame of the blowpipe. What takes place? The orange incrustation, turning yellow when cold, is bismuth oxide.

b. Warm 0.5 g. of bismuth with dilute nitric acid. What product is formed?

c. Add sodium hydroxide, drop by drop, to 10 ml. of a solution of bismuth nitrate until precipitation is complete. Filter off the precipitate, wash it with water, and use it in *d*, *e*, and *f*.

d. Remove a small portion of the product from *c* and add sodium hydroxide solution. Does the substance dissolve?

e. To another portion of the product from *c* add dilute hydrochloric acid. What property is hereby illustrated?

f. Pour through the filter containing the remainder of the product from *c* a solution of sodium stannite. What type of reaction takes place?

g. Add a few drops of bismuth nitrate solution to 10 ml. of water. What is the product? Determine whether or not this reaction is reversible.

133. Preparation of Basic Bismuth Nitrate. Dissolve 10 g. of bismuth nitrate in 3 ml. of dilute nitric acid and 5 ml. of water by shaking the mixture in a small stoppered flask. Pour the solution obtained into 500 ml. of water and stir the mixture for two or three minutes. Filter off the precipitate on a suction filter, wash it at once with a small amount of water, and dry it.

EXERCISES

1. Make a table showing in separate columns the most important metallic and nonmetallic properties of each of the elements studied in this chapter.

2. What is the relationship, in this group, between the atomic weight of the element and the stability of its hydride?

3. Draw a parallel between the chemical nature of the oxides and the sulfides of arsenic and antimony.

CHAPTER XXVII

CARBON

134. Preparation of Wood Charcoal. Fill a hard-glass test tube about half full of hard-wood sawdust and clamp it in position as shown in Fig. 55. Provide the test tube with a delivery tube connected with a test-tube receiver which is fitted with a glass jet. Place the receiver in a beaker of water. Heat the wood gently at first and then strongly. Apply a light to the gases escaping from the jet. Are they combustible? Continue the heating of the sawdust until no more gases are evolved. Examine the liquid in the receiver. What is its odor? its reaction toward litmus? Is it combustible? Is it soluble in water? Examine the charcoal in the hard-glass test tube and list its properties.

Pulverize some freshly heated charcoal and place it in a filter in a funnel. Pour through it very dilute solutions of litmus and potassium permanganate. What is your observation?

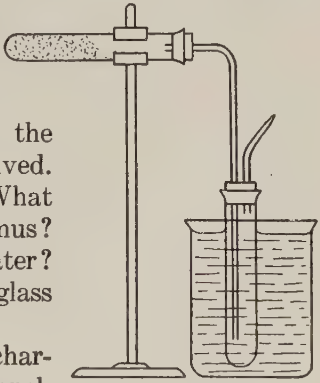

FIG. 55

135. Preparation of Coke. Repeat the preceding experiment, substituting powdered soft coal for the sawdust. What differences do you observe in the liquid in the receiver?

136. Preparation of Lampblack. Hold a porcelain dish in the luminous flame of a Bunsen burner. Account for the deposit of carbon. Why is it so smooth and so adherent? Are its particles large or small? Why?

137. Reduction with Carbon. Mix 0.5 g. of powdered charcoal with twice its weight of litharge (PbO). Mix the two thoroughly, and heat them strongly in a crucible. Examine the product left in the crucible. What is it? Account for its formation.

138. Graphite. Examine some graphite and list its properties. Does it burn as readily as lampblack does? Compare their resistance to the action of concentrated nitric acid.

139. Carbon Dioxide. *a*. Place in a generating flask 2 g. of powdered marble or limestone. Add, through the thistle tube, dilute hydrochloric acid. Collect two bottles of the gas by the upward displacement of air. Into one of the bottles introduce a burning splint. What is the result? Shake the contents of the other bottle with 10 ml. of limewater ($Ca(OH)_2$). What is the precipitate? Give the equation for its formation. Is carbonic acid active or weak? stable or unstable? Give five other ways of preparing carbon dioxide.

b. Lead some carbon dioxide from the generator into 2 ml. of limewater. Continue to pass the gas into the solution for a minute or two. Explain your observations. Give equations.

c. Blow your breath through limewater for a minute or two. Are the results similar to those obtained with carbon dioxide? What is your conclusion?

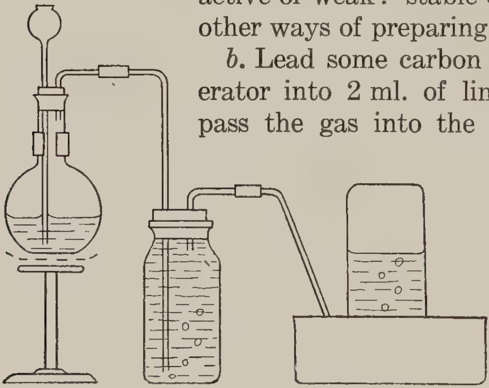

FIG. 56

140. Carbon Monoxide. Arrange an apparatus as shown in Fig. 56. Place in the generating flask 2 g. of oxalic acid and add 5 ml. of concentrated sulfuric acid through the thistle tube. Heat the mixture cautiously. Lead the gas through a wash bottle containing sodium hydroxide, and collect the carbon monoxide over water. Apply a flame to a bottle of the gas. Note the color of the burning gas. What are the physiological effects of carbon monoxide? What is the purpose of sodium hydroxide?

141. Carbonates. Try the action of dilute sulfuric acid on the carbonates of sodium, potassium, magnesium, and lead. Formulate for magnesium carbonate the ionic equilibria showing why this reaction goes nearly to completion. How could you show the presence of carbonates in the soil?

EXERCISES

1. How may it be proved that pure graphite and pure diamond are carbon?

2. Why is soft coal and not anthracite used in making coke?

3. Does the physical condition of animal charcoal make it more suitable than lampblack as a decolorizing agent? Which would be the more suitable for making ink?

4. Suggest some ways by which water gas may be separated into hydrogen and carbon monoxide.

5. Explain why carbon monoxide is such a valuable fuel.

6. Which would produce the more heat: burning 10 g. of charcoal to carbon dioxide or burning 20 g. of carbon monoxide to carbon dioxide?

CHAPTER XXVIII

THERMOCHEMISTRY

142. Heat of Neutralization. Use the calorimeter described in Experiment 73 (Fig. 41). Prepare 150 ml. of a 0.2-*N* hydrochloric acid by diluting the concentrated acid (12-*N*) found in your desk. Obtain 150 ml. of a 0.2-*N* sodium hydroxide solution and pour it into the inside container of the calorimeter. Read the temperature accurately. Pour the acid solution into the base and stir the mixture. Note and record the highest temperature reached. Assume that 12 calories of heat are absorbed by the calorimeter and that the specific heat of the solution is 1. Calculate the calories of heat evolved from the amounts of acid and base used. Calculate the amount of heat that would be liberated if one gram equivalent of the acid had reacted with one gram equivalent of a base. Why should you expect that the heat of neutralization of all active acids and bases would be nearly the same? Compare this result with that in Experiment 90.

143. Heat of Dilution. Place 100 ml. of sulfuric acid (1 part water to 1 part concentrated sulfuric acid) in the inner container of the calorimeter. Read its temperature and add 10 ml. of water at the same temperature as the acid. Stir the solution thoroughly and note the highest temperature. How many calories of heat were produced by dilution? (Assume a specific heat of 1.)

EXERCISES

1. Explain why no perceptible heat change is noted when dilute solutions of sodium chloride and potassium nitrate are mixed.

2. The heat of formation of carbon dioxide is 97,000 cal., the heat of formation of carbon monoxide is 29,000 cal. How many calories of heat will be formed when 2000 g. of carbon monoxide are burned?

3. Define the term "B.T.U."

4. Given the following thermochemical equations calculate the heat of formation of K_2SO_4:

$$2\,K + \tfrac{1}{2}\,O_2 \longrightarrow K_2O + 98{,}200 \text{ cal.}$$
$$S + 1\tfrac{1}{2}\,O_2 \longrightarrow SO_3 + 91{,}900 \text{ cal.}$$
$$K_2O + SO_3 \longrightarrow K_2SO_4 + 154{,}200 \text{ cal.}$$

5. Given the following thermochemical equations, calculate the heat of combination of MgO and SO_3:

$$Mg + \tfrac{1}{2}\,O_2 \longrightarrow MgO + 143{,}400 \text{ cal.}$$
$$S + 1\tfrac{1}{2}\,O_2 \longrightarrow SO_3 + 91{,}900 \text{ cal.}$$
$$Mg + S + 2\,O_2 \longrightarrow MgSO_4 + 300{,}900 \text{ cal.}$$

CHAPTER XXIX

THE FURTHER CHEMISTRY OF CARBON

144. Preparation of Methane. Mix from 2 to 5 g. of powdered sodium acetate with twice its weight of soda lime. Place the mixture in a hard-glass test tube fitted with a cork bearing a glass delivery tube so arranged as to dip under water in a pneumatic trough (Fig. 20). Collect test tubes full of the gas over water. (Do not collect the gas until it is bubbling freely. Why?) Bring a lighted splint to the mouth of one test tube. What is the result? Write the equation. To another test tube add 1 ml. of bromine water, and stopper tightly. Note any change in color in one minute; in twenty minutes; at the end of the laboratory period.

145. Preparation of Ethylene. Fit a delivery tube and a thistle tube to a 250-milliliter generating flask (Fig. 56). Place a little dry sand in the flask and add 5 ml. of alcohol and 12 ml. of concentrated sulfuric acid. Connect the delivery tube with a wash bottle half full of sodium hydroxide solution. Fit the wash bottle with a delivery tube arranged to lead the gas into a pneumatic trough. Heat the generator, and then add slowly and gradually through the thistle tube a cooled mixture of 1 vol. of alcohol to 2 vols. of concentrated sulfuric acid.

Collect two test tubes full of gas by displacement of water. Note its physical properties. Burn the gas in the tube. Write the equation. Add 1 ml. of bromine water to one test tube. Stopper the tube at once and shake it. How soon does the color disappear? Why?

146. Preparation of Iodoform. Dissolve 0.5 g. of potassium iodide in 10 ml. of water in a 100-milliliter flask and add 1 ml. of acetone. Add to the mixture, through a separatory funnel, a solution of sodium hypochlorite, shaking the flask after each addition until no further precipitate forms. Allow the precipitate to settle, decant off the liquid, wash the residue with water two or three times, filter it, and dry it on filter paper. Recrystallize the compound from alcohol. To bring the iodoform into solution, heat the water in a water bath above the boiling-point of alcohol, *turn out the flame*, and then place a flask containing the iodoform and alcohol on the water bath, putting in more alcohol, if necessary. If not clear, filter the solution before placing it in a clean dry crystallizing dish. Set the dish away in a cool place and allow the crystals to separate out gradually without further heating.

Determine the properties of iodoform: melting-point, solubility in water, alcohol, carbon disulfide, crystal form, taste, odor, color, reaction with silver nitrate, etc. In what way do the organic halogen compounds differ from inorganic halides?

147. Preparation of Formaldehyde. (Catalytic oxidation of methyl alcohol.) This experiment is to be performed by two students working together.

The apparatus for this experiment consists of a small distilling flask to the side tube of which is attached, by means of a cork stopper, a piece of *hard-glass tubing* which is bent in the middle very slightly and so arranged that the bend extends upward. The other end of the hard-glass tubing is closed by a cork stopper carrying a delivery tube which leads through a cork and extends nearly to the bottom of an empty wide-necked flask. Through a second opening in this cork pass a short tube bent at a right angle. The neck of the distilling flask carries a cork through which passes a glass tube reaching to the bottom of the flask (Fig. 57).

Place 25 ml. of methyl alcohol and 5 ml. of water in the distilling flask, and in the middle of the hard-glass tube *A* place a layer of oxidized copper turnings (to oxidize the copper turnings, hold them in a pair of crucible tongs and move the turnings about in a Bunsen flame for a few seconds). Then make sure that the whole apparatus is air-tight by attaching a tube to the exit end and blowing gently into it. This will cause the alcohol in the distilling flask to rise in the glass tube; and if the exit tube is closed, the level of the alcohol in the tube will not fall, provided the apparatus does not leak. When the apparatus is air-tight, surround the wide-necked flask with a freezing-mixture of ice and salt and connect the exit tube with the suction.

Heat the distilling flask on a water bath the temperature of which is about 65°. Heat the copper turnings to redness, using a wing-top burner, and then draw a slow stream of air through the apparatus until about three fourths of the alcohol is volatilized. Heat the copper only enough to start the reaction. As soon as it starts to glow within the tube, do not heat it any more.

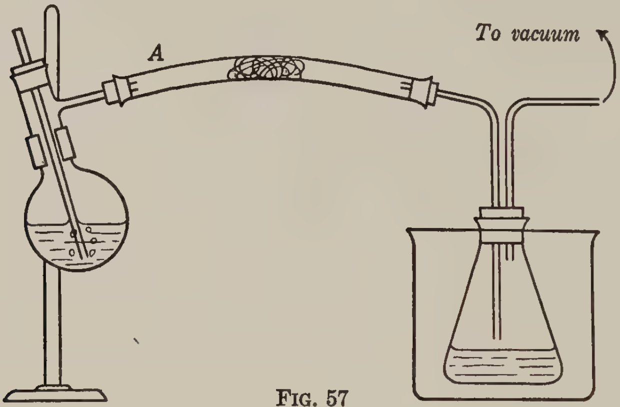

FIG. 57

Notice that the heat of the reaction keeps it glowing. To stop the reaction, *remove* the water bath and then shut off the suction *very gradually.*

The liquid which condenses in the bottle will contain about 40 per cent of formaldehyde. Water, methyl alcohol, and formic acid are the other substances present. Filter the liquid and use it in Experiment 148.

148. Properties of Formaldehyde. *a.* Evaporate about 5 ml. of the liquid in an evaporating dish over the water bath. What is left? Write the equation.

b. Dilute 1 ml. of silver nitrate solution to 10 ml. with water. To this add very dilute ammonium hydroxide, drop by drop, until the precipitate which first forms just redissolves. Similarly dilute 1 ml. of the liquid containing the formaldehyde to 10 ml. with water. Place 1 ml. of the ammoniacal silver nitrate solution in a test tube, add 5 ml. of water, shake, add 1 ml. of the diluted formaldehyde solution, shake again, and place the test tube containing the mixture in a beaker of boiling water. Describe the result and write the equation.

c. Put 1 ml. of the diluted formaldehyde solution into a test tube and add to it 5 ml. of water. Shake the mixture, add to it a few drops of 1-per-cent solution of phloroglucinol, and then make the solution slightly alkaline with dilute sodium hydroxide. Describe the result.

d. Mix 5 ml. of the formaldehyde solution with 5 ml. of concentrated ammonia in a small evaporating dish. Evaporate the solution to dryness on the water bath. Dissolve the solid in 10 ml. of boiling alcohol (boil it on the water bath, not over a free flame), filter while hot, and cool the filtrate. Then filter off the crystals which result. Dry the crystals carefully. The solid is hexamethylene tetramine or "urotropin." It is used medicinally as a urinary antiseptic; chemically, in the preparation of synthetic resins of the bakelite type; for absorbing poisonous gases in gas masks; as an accelerator (catalyst) in the vulcanization of rubber; etc. It sublimes when heated, and is very soluble in water. Why was the hard-glass tube (Experiment 147) bent in the middle, and why was this part placed higher than the ends of the tube? Why have the apparatus air-tight? Why pass air through the apparatus along with the alcohol vapor?

149. Preparation of Oxalic Acid. Into a Florence flask with a capacity of about 1 liter place 25 g. of cane sugar and 125 g. of nitric acid (specific gravity 1.42) and heat the mixture gently on a water bath under the hood. Gases are evolved. Explain. When the reaction has ceased, evaporate the liquid to about one quarter its bulk. On cooling, colorless crystals of oxalic acid separate out. Drain them through glass wool and recrystallize them from a small amount of distilled water.

Determine the properties: color, solubility, crystalline form. Does it sublime? Is it a reducing agent? Heat some oxalic acid with a small amount of concentrated sulfuric acid and prove that both oxides of carbon are given off.

To a solution of the acid add a few drops of dilute sulfuric acid, and warm the mixture gently. On adding permanganate solution, it is immediately decolorized. What has taken place?

150. Preparation of Ethyl Acetate. Place about a gram of sodium acetate in a test tube. Add 2 ml. of ethyl alcohol and two or three drops of concentrated sulfuric acid. Warm the mixture gently, and note the fruity odor. What is the function of the acid? If ethyl acetate were boiled in water containing dilute hydrochloric acid, what would be formed?

151. Preparation of an Ether. (Methyl beta-naphthyl ether (Jara-Jara).) Add 10 drops of methyl alcohol to 0.1 g. of beta-naphthol ($C_{10}H_7OH$) and then add two drops of concentrated sulfuric acid. Warm the mixture gently, and note the odor. This ether is used for preparing cheap perfumes. Its formula is $C_{10}H_7OCH_3$. The ethyl beta-naphthyl ether has an odor which resembles orange blossoms. What is the formula for diethyl ether? For what is it used?

CHAPTER XXX

SILICON

152. Properties of Silicon. List the physical properties of silicon. Make a solution of 8 g. of sodium hydroxide in 10 ml. of water. Add 1 g. of powdered silicon, heat the mixture slowly, and test the gas evolved with a lighted splint. Does it burn? What is this gas?

153. Properties of Silica. (*Optional*) Grind 0.5 g. of sand with five times its weight of a mixture of equal parts of sodium and potassium carbonate. Place the mixture in a platinum crucible and heat it with the blast lamp until fusion results. Cool the crucible and contents and immerse them in hot water. Remove the crucible when the material has disintegrated. Filter the solution, if necessary. Transfer the solution to an evaporating dish and make it acid with hydrochloric acid. Evaporate the mixture to dryness (*CARE!*) and heat the dish and residue for one hour at approximately 125°. Cool the dish, and add 3 ml. of hydrochloric acid and 20 ml. of water. Warm the dish, and pour off the clear solution. Wash the residue several times with water by decantation. Examine the residue carefully. What is it?

154. Silicic Acid. Add 5 ml. of concentrated hydrochloric acid to 5 ml. of sodium silicate solution. Explain your observation. Ignite this material in an evaporating dish. What is formed?

155. Properties of Glass. Heat a piece of soft-glass tubing in the Bunsen flame until it softens and sags perceptibly under its own weight. Repeat, but use a piece of hard-glass tubing. Does it sag as readily as the soft glass did?

156. Properties of Cement. Place 1 g. of Portland cement in a crucible. Add small amounts of water, and stir the mixture after each addition. When the mixture is of about the consistency of thick mud, set it aside for several days. Examine the resultant material. What has taken place? Repeat this test, using 0.5 g. of cement and 1.5 g. of fine sand. After several days examine the resultant product. Can you distinguish the sand grains? What holds them together?

EXERCISES

1. What is *ferrosilicon*? For what is it used?

2. What is *duriron*? What are some of its uses?

3. List some of the silicon halides with their carbon analogues.

4. What is *water glass*? List several of its uses.

5. What types of raw materials are used in making Portland cement?

CHAPTER XXXI

BORON

157. Properties of Borax. *a.* Test the effect of a borax solution on litmus. Explain your observations. Heat a few crystals of borax in a hard-glass test tube. Does water condense on the upper part of the tube? Explain. Test the action of a solution of silver nitrate with concentrated and with dilute borax solutions. Explain any differences observed. Write all equations.

b. Add 3 ml. of alcohol to 2 ml. of a solution of borax. Place the solution in an evaporating dish and add three drops of sulfuric acid. Set fire to the alcohol. What color is the flame?

158. Preparation of Boric Acid. Dissolve 5 g. of borax in 15 ml. of hot water. Add to the hot solution 5 ml. of concentrated hydrochloric acid. Filter off the precipitate which forms when the solution is cooled. What is it?

159. Borax-Bead Tests. Make a small loop on the end of a platinum wire. Heat the wire in the Bunsen flame and dip it into finely powdered borax. Hold the borax in the oxidizing flame until it fuses to a clear transparent bead. Touch the clear colorless bead to a very small portion of cobalt oxide. Again hold the bead in the flame until it is transparent. Note carefully the color of the bead when hot and when cold. If the bead is dark or opaque, either too much material has been used or more heating is needed. If more heating does not give a transparent bead, a new bead must be made and the test be repeated. Repeat the test, using oxides of nickel and copper.

EXERCISES

1. Give the name and the formula of the acid related to borax.

2. Why is borax often used in the household for treating water? Explain clearly, giving equations.

3. List some uses of boric acid.

4. What property of borax makes it useful in the welding of iron?

CHAPTER XXXII

THE METALS

160. Colors of the Common Metals. Polish the surfaces of pieces of copper, zinc, iron, magnesium, aluminum, silver (use a coin), and bismuth. Give the color of each metal.

161. An Alloy of Lead and Tin. Melt 1 g. of lead in a crucible, and add to it 1 g. of tin. What is the common name of the alloy? Observe whether the product is harder or softer than lead; than tin.

162. Hydrolysis. Treat 0.5 g. of bismuth oxide with 3 ml. of concentrated hydrochloric acid. Does the oxide dissolve when the mixture is stirred? Dilute the clear liquid with from ten to twenty times its volume of water. What is the precipitate? Is it soluble in hydrochloric acid? Is bismuth a metal or a nonmetal?

Try the solubility of silicon dioxide in concentrated hydrochloric acid. What should you expect to happen if silicon tetrachloride were treated with water? Should you expect the action to be reversible? Is silicon a metal or a nonmetal?

Recall the behavior of the halides of phosphorus with water. Were these hydrolytic reactions reversible?

EXERCISES

1. What is the reason that sodium never occurs free in nature?

2. Why does so active an element as oxygen occur free in nature?

3. From your study of arsenic, outline the reasons for calling it a nonmetal? Does it possess any property which is characteristic of the metallic elements?

4. When nitric acid acts on carbon, carbon dioxide is produced. Is this a general method for preparing oxides? What other oxides may be prepared in this way? What is the usual product of the action of this acid on metals? on nonmetals?

5. Given nitrogen, hydrogen, and oxygen, how should you prepare ammonium nitrate?

CHAPTER XXXIII

THE METALS OF THE ALKALIES

163. Properties of Sodium. *a.* Give in tabular form the physical properties of metallic sodium: color, hardness, etc.

b. Obtain a piece of sodium about the size of a pea from the storeroom. Cut it into three unequal pieces. Allow the smallest piece to stand on a watch glass. What happens to the shiny metallic surface? When the sodium has apparently disappeared, test the product with dry litmus paper. What is the result? Burn the largest piece of sodium in a deflagrating spoon. Note the color of the flame. What is the product formed? Devise and carry out an experiment to establish the correctness of your prediction.

c. Place the other piece of sodium in a beaker containing a small amount of water, and immediately cover the beaker with a watch glass. Account for the behavior of the sodium. When the action has ceased, test the solution in the beaker with litmus paper. Would this be a practical way of preparing the substances formed? In what other ways may sodium hydroxide be prepared?

164. Preparation of Sodium Hydrogen Carbonate. Add somewhat more than enough salt (about 30 g.) to saturate 100 ml. of 5-N ammonium hydroxide. Prepare the mixture at least an hour before it is to be used, or preferably let it remain in your desk until the next laboratory period. Decant the clear liquid into an Erlenmeyer or Florence flask which has been fitted with a one-hole stopper and a glass tube (Fig. 26). Adjust the tube until the end is about half an inch above the surface of the solution, and connect with a carbon dioxide (Kipp) generator. Remove the stopper for a few moments in order that the air may be displaced, then replace the stopper. Cool the mixture by letting the flask stand in cold water, and shake it occasionally. When no more precipitate is formed, disconnect the flask and decant the liquid into a beaker. Wash the sodium bicarbonate with a little cold water, and again decant the liquid, letting as much liquid flow off as possible without loss of appreciable solid bicarbonate.

Write equations for all reactions involved in the preparation of sodium bicarbonate. What does the solution that was decanted from the hydrogen carbonate contain? What disposal would be made of this solution in a plant producing $NaHCO_3$?

165. Properties of Sodium Hydrogen Carbonate (Sodium Bicarbonate). *a.* Place the sodium hydrogen carbonate which you prepared, or about 5 g. of the substance from the side shelf, in a hard-glass test tube and heat it cautiously until all the water is driven off, and then more strongly. What gas is given off? Carry out tests to verify your conclusion. What is the solid that remains in the tube? Is this reaction typical of all bicarbonates?

b. Dissolve 0.5 g. of sodium hydrogen carbonate in 10 ml. of water. Divide the solution into two parts. To one add a drop of phenolphthaleïn. Test the second with red and with blue litmus paper, and then add a drop of methyl orange. What does this experiment indicate as to the sensitiveness of the three indicators to the presence of OH ion?

c. Prepare a dilute solution of sodium carbonate. Divide it into two parts, and test with the indicators used above. Account for the difference between the results in the two cases.

d. State two methods of distinguishing between bicarbonates and carbonates.

166. Baking Powder. *a.* Mix a little cream of tartar (potassium hydrogen tartrate) with some sodium bicarbonate. Add enough water to make a paste. What gas is evolved?

b. Pulverize a little primary sodium phosphate and mix with a little sodium bicarbonate; then add enough water to make a paste. What gas is evolved?

c. Repeat *a,* using potassium aluminum sulfate instead of cream of tartar. Account for your observation. These experiments illustrate the three types of baking powder commonly used. Cornstarch is also added to keep the mixture dry.

d. Account for the use of sodium bicarbonate with sour milk as a substitute for commercial baking powders.

167. Preparation of Pure Sodium Chloride. Add about 10 per cent more of impure salt than is required to saturate 50 ml. of water, and boil the mixture until nearly all the solid is dissolved. Cool, and filter to remove any foreign matter. Add concentrated hydrochloric acid slowly until no more precipitate forms. Then decant off the mother liquor. Wash the salt once or twice by decantation, using cold water. Finally drain off as much water as you can, and dry the salt between pieces of filter paper.

Write equations to show the equilibrium between sodium chloride with its ions and hydrogen chloride with its ions. Explain clearly but concisely why the hydrochloric acid precipitates the sodium chloride and not the magnesium chloride, which is a common impurity in the salt. Why is magnesium chloride objectionable in salt?

168. Properties of Sodium Hydroxide. *a.* Add some sodium hydroxide solution to an ammonium salt, and warm. Note the odor. Give an equation to show the reaction taking place. Indicate the gas formed.

b. Add a few drops of a solution of $NaOH$ to solutions of $CaCl_2$, $CdCl_2$, and $Pb(NO_3)_2$. Write equations for the reactions which take place, and indicate the precipitates formed in each case.

c. Add a large excess of sodium hydroxide to each of the solutions used above. Explain your results. What other hydroxides react similarly to lead hydroxide in this respect?

d. Pour a solution of sodium hydroxide into a test tube containing a small piece of woolen material. Warm. Repeat, using cotton. What practical use may be made of this property of $NaOH$?

169. Equivalent Weight of Sodium. Place a short-stemmed funnel in a pneumatic trough nearly full of water (Fig. 58). Invert a 100-milliliter Erlenmeyer flask full of water over the funnel by tipping the funnel on its side until the stem is under the water; then bring both to a vertical position without allowing any air to get into the system. Let the lip of the flask rest on the sides of the funnel, and draw off water until the level is about half an inch above the lip of the flask. Close a piece of glass tubing having a diameter of about 4 mm. and cut it to a length of 5 cm. When the tube is cool, weigh it accurately, and then place in it about 0.15 g. of small pieces of sodium having bright surfaces. The kerosene should be removed from the sodium by pressing between pieces of filter paper. Press the sodium into one compact mass with a glass rod. Accurately weigh the tube and contents, and then place a plug of cotton in the open end of the tube, allowing a little to protrude. Hold the tube containing the sodium with iron forceps or crucible tongs, tip the funnel, and place the tube beneath it. Remove the cotton by means of forceps. When the action has ceased, hold the flask by that portion of the neck below the water, and equalize the levels of the water inside and outside of the flask. Mark the flask at the level of the water, remove it, and measure the volume of hydrogen evolved. Note the temperature of the hydrogen and the barometric pressure. From your data calculate the weight of the hydrogen in the dry condition and the equivalent weight of sodium. Since the valence of sodium is 1, this weight is also the gram atomic weight of sodium. All data are to be recorded in tabular form.

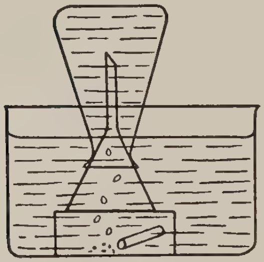

FIG. 58

170. Properties of Potassium. Place a small piece of potassium on some water. What substances are formed as a result of the reaction? Devise experiments to test your conclusions.

171. Properties of Potassium Hydroxide. Repeat Experiment 168 *b*, using KOH instead of NaOH. Can these two substances be used interchangeably?

172. Preparation of Potassium Nitrate. Dissolve 22 g. of potassium chloride in 45 ml. of water and add 25 g. of sodium nitrate. Boil the mixture until the volume has been reduced to two thirds of the original volume. Then stop heating the mixture, but let it stand on the hot wire gauze until the precipitate settles. Decant the clear liquid into a second beaker. Cover the beaker with a piece of filter paper and set it aside to cool.

Write the equation for the reaction and indicate the nature of the precipitate formed in the hot solution. Give the solubility of each of the salts at 18° and at 100°. Decant the liquid. Are the crystals of the same material as those formed above? A second crop may be obtained by evaporating the mother liquors to half their volume, and cooling.

What use of potassium nitrate led to this method of preparation? What property of sodium nitrate prevents its use for the purpose?

173. Special Reactions of the Ions of Sodium, Potassium, and Ammonium. *a.* Test solutions of $NaCl$, KCl, and NH_4Cl with each of the following reagents: saturated solutions of picric acid, sodium hydrogen tartrate, perchloric acid, sodium cobaltinitrite, potassium pyroantimonate.

Write equations to show the products formed; indicate any precipitates, and state their colors.

Supposing a solution contained only Na^+ or K^+, how could you tell which it contained by the use of any of the reagents used above?

Can tests for K^+ or Na^+, using the reagents given above, be made in the presence of NH_4^+? Give reasons for your answer.

b. Hold a piece of nichrome or preferably platinum wire in a nonluminous flame until no color is imparted to the flame. Then moisten it with a solution of $NaCl$. After noting the color imparted to the flame, observe the flame through a flat bottle containing a saturated solution of chrome alum. Account for the difference in your observations.

Repeat, using a solution of KCl and of NH_4Cl.

c. Now moisten the wire with a solution containing a mixture of KCl and $NaCl$, and heat. How can you detect the presence of K^+ in a mixture of Na^+ and K^+ by the use of the chrome-alum filter? (The chrome-alum filter will filter out light of the wave lengths imparted to a flame by the ions of Na, Ca, Ba, Sr.)

EXERCISES

1. From the position of rubidium and cæsium in the periodic table, what can you say as to their probable valence, activity, and other properties?

2. How many grams of coke containing 90 per cent of carbon would be required to prepare 1060 lb. of sodium carbonate?

3. What volume of CO_2 can be prepared from 8.4 g. of $NaHCO_3$?

4. With how many equivalent weights of sulfuric acid will 0.4 g. of sodium hydroxide react?

5. Starting with $CaCO_3$, give equations showing how to prepare K_2CO_3.

6. Starting with ferrous sulfate, give equations to show how ferric hydroxide might be prepared.

7. Give equations to show what happens at the electrodes when a solution of NaCl is electrolyzed.

8. Account for the difference in activity of sodium and potassium in terms of electrons and their arrangement.

CHAPTER XXXIV

COPPER, SILVER, AND GOLD

174. Copper. *a.* Form a helix by winding 10 cm. of 18-gauge copper wire round a lead pencil. Take the helix off the pencil, grasp one end of the wire with your crucible tongs, and heat the wire in the oxidizing portion of a Bunsen flame. Remove the wire before it melts, and note any change in its appearance. What is the melting-point of copper? What is the coating on the wire? Now heat the wire to redness, and quickly drop it into a test tube containing 5 ml. of methanol (wood alcohol). What takes place? Describe the changed appearance of the wire and note the odor of the gaseous oxidation product of the alcohol. What is the gas? Recall the ease of reduction of cupric oxide by hydrogen (Experiment 26).

b. Test separately the action of dilute and concentrated hydrochloric, nitric, and sulfuric acids on small pieces of copper. If no action occurs in the cold, try the effect of heat. Is hydrogen evolved in any of the cases? How should you have been able to predict the answer to the foregoing question without carrying out the experiments? Write equations for all reactions taking place.

175. Cuprous Compounds. *a.* Dissolve 5 g. of cupric chloride in 20 ml. of water in a 100-milliliter beaker. Add 20 ml. of concentrated hydrochloric acid together with 2.5 g. of copper wool. Boil the mixture vigorously (*HOOD!*) until the greenish color of the solution changes to a clear amber. The color change should occur after boiling for about two minutes. To what ion is the greenish tint due? What is indicated by the disappearance of this color? Write an equation for the reaction that takes place. What is the function of the metallic copper?

Reserve 5 ml. of the solution for use in *b*, and pour the remainder into a beaker containing 200 ml. of water. What is the precipitate? Allow it to settle, and then decant the clear liquid above the solid. To the latter add 50 ml. of water, stir thoroughly, and filter. Wash the precipitate on the filter by pouring through it a mixture of 23 ml. of water and 2 ml. of concentrated hydrochloric acid. Divide the washed cuprous chloride into three equal parts.

Place one portion of the moist cuprous chloride in a test tube, and wash the salt down into the bottom of the test tube with approximately 5 ml. of water. Quickly cover the water with half its volume of kerosene, and add 3 ml. of concentrated ammonium hydroxide. Stir the solution carefully with a small glass rod until all the salt dissolves. If any remains undissolved, add more ammonium hydroxide. Note the color of the aqueous layer. Bubble air through the ammoniacal solution. Note the deepening of the color. Write the equation for the action of ammonium hydroxide on cuprous chloride. What is the effect of air on the solution? Why was the kerosene added?

Add concentrated hydrochloric acid to another portion of the cuprous chloride. Write an equation for the reaction that takes place. Explain the solubility of cuprous chloride in ammonium hydroxide and in hydrochloric acid.

Save the third portion of the cuprous chloride for use in Experiment 178, *b*.

b. To 5 ml. of the solution of cuprous chloride prepared in *a* add a slight excess of sodium hydroxide solution. What is the precipitate? Write an equation for the reaction. After the precipitate has settled, decant the supernatant liquid, add 10 ml. of water to the precipitate, and filter it off. Wash the solid once more by pouring 20 ml. of water through the filter.

Take a small portion of the cuprous oxide and heat it in a test tube with 10 ml. of sodium hydroxide solution. Is the oxide amphoteric?

Test separately the action of small portions of cuprous oxide with concentrated hydrochloric acid and with ammonium hydroxide. Note results, and write an equation for each reaction. Explain the solubility of cuprous oxide in the inactive base ammonium hydroxide. Shake the solution of ammoniocuprous hydroxide with air. How is the formation of another ion indicated? What is the ion? Compare Experiment 176, *e*.

Suspend a small quantity of cuprous oxide in 5 ml. of water in a test tube, and shake persistently for five minutes, taking care to admit fresh air frequently. Is there any change in the appearance of the oxide? Now add 5 ml. of ammonium hydroxide. To what extent was cuprous oxide affected by the air? Draw conclusions as to the stability of cuprous oxide in the presence of air and moisture.

c. Add 2 ml. of glucose solution to 10 ml. of Fehling's solution (Reference). Bring it slowly to the boiling-point. What takes place? Refer to your text for the value of Fehling's solution as a test reagent.

d. Add potassium iodide solution, drop by drop, to 10 ml. of a solution of cupric sulfate until precipitation is complete. Note carefully what occurs. Add an excess of 2 ml. of potassium iodide solution, filter off the precipitate, and wash it *thoroughly* with water. Do not allow the washings to run into the original filtrate. Dilute 1 ml. of the latter with 10 ml. of water and add 3 ml. of carbon disulfide. What is extracted by the carbon disulfide? To 1 ml. of the original filtrate, diluted with 10 ml. of water, add a little freshly prepared starch solution. Confirm your answer to the preceding question. Note the color of the solid on the filter paper and tell what the substance is. Write equations to express the steps by which its formation takes place. What can you say as to the stability of cupric iodide?

176. Reactions of Cupric Compounds. Prepare 24 ml. of dilute cupric sulfate solution by adding 12 ml. of water to an equal volume of the cupric sulfate solution found on the reagent shelf. For each of the following tests use 3-milliliter portions of the solution you have just prepared.

a. What takes place on the addition of several drops of sodium hydroxide solution to dilute cupric sulfate? Write the equation. Now add 5 ml. more of the sodium hydroxide solution. Is the precipitate soluble in an excess of the base? Boil, and note what occurs. Write the equation.

b. Add a few drops of a solution of potassium ferrocyanide to another portion of the dilute cupric sulfate solution. Note results, and give equation for the reaction. Place in a large test tube one drop of the cupric sulfate solution found on the reagent shelf and 50 ml. of water. Test this solution with potassium ferrocyanide solution. What is the result? This delicate test serves for the detection of 1 part of copper in 200,000 parts of solution.

c. Drop a small piece of aluminum into a test tube containing a 3-milliliter portion of dilute cupric sulfate solution. Explain the result, and write an ionic equation to express the reaction. Determine in like fashion the action of zinc and of tin. Explain results.

d. Test separately 3-milliliter portions of dilute cupric sulfate solution with hydrogen sulfide and with colorless ammonium sulfide. Write an equation for each reaction. Filter off both precipitates on the same filter paper, and wash the combined precipitate with water until the odor of hydrogen sulfide is no longer perceptible. Reserve a small portion of the cupric sulfide for use in Experiment 178, *c*, and test separately the solubility of small amounts of the remainder in dilute and concentrated hydrochloric, sulfuric, and nitric acids. If action does not occur in the cold, try the effect of heat. Tabulate your results. Is cupric sulfide soluble in ammonium hydroxide?

e. Add one drop of ammonium hydroxide to 3 ml. of dilute cupric sulfate solution. Is the precipitate the same substance as that formed in the first part of *a*? Write the equation. Now add an excess of ammonium hydroxide. Note results and write an equation to express the reaction. Explain.

f. Clean a platinum wire carefully, and note the flame reaction given by a solution of cupric chloride. Again clean the wire, until it does not color the flame. Test the flame reaction of a solution of cupric sulfate. Only the halides of copper can be used in making a flame test for the element.

177. Preparation of Ammoniocupric Sulfate. Pulverize 10 g. of cupric sulfate crystals and dissolve the powder in a solution of ammonium hydroxide prepared by the addition of 10 ml. of water to 15 ml. of *concentrated* ammonium hydroxide. If the resulting deep-blue solution contains any insoluble material, it should be filtered through a thin mat of asbestos wool placed in a funnel (*instructor*). Now stir the clear solution vigorously while slowly adding to it 15 ml. of 95-per-cent alcohol. Bluishpurple crystals of ammoniocupric sulfate, $Cu(NH_3)_4SO_4 \cdot H_2O$, precipitate out. Explain. Set these aside for an hour, and then filter. Wash the crystals by first pouring through the filter 20 ml. of a mixture of equal volumes of 95-per-cent alcohol and concentrated ammonium hydroxide, and then 10 ml. of alcohol alone. By the use of a spatula transfer the compound from the filter paper to a watch glass, and allow the crystals to dry at the temperature of the laboratory. Note carefully the color of the crystals, and set them aside in your desk for examination at the next laboratory period. Is a color change noticeable? What would a color change indicate? Does the compound lose ammonia on exposure to air at ordinary temperatures? What can you say as to the stability of the salt under these conditions? Try its solubility in water containing a little ammonium hydroxide.

178. Complex Copper Cyanides. *a.* Dilute 2 ml. of the cupric sulfate solution on the reagent shelf with an equal volume of water. Add a drop of potassium cyanide solution (*CARE, POISON!*). What is the precipitate? Now add an excess of potassium cyanide solution. Note results, and write equations for the reactions that have occurred.

b. Test the action of potassium cyanide solution on cuprous chloride (from Experiment 175, *a*). Write an equation to express the reaction. Cuprous chloride is very insoluble in water. How do you explain its solubility in potassium cyanide solution?

c. Treat cupric sulfide (from Experiment 176, *d*) with potassium cyanide solution. What result is observed? Write an equation for the reaction.

d. Prepare a solution of ammoniocupric sulfate by the addition of an excess of ammonium hydroxide to 3 ml. of a solution of cupric sulfate. Now add potassium cyanide solution, drop by drop. Write an equation for the reaction. Why does it go to completion?

179. Preparation of Silver Nitrate. (*Two students working together.*) Place a small piece of sterling silver or a dime in an evaporating dish and add 20 ml. of nitric acid prepared by diluting 10 ml. of the concentrated acid with an equal volume of water. Warm gently (*HOOD!*) until a vigorous reaction sets in.

Why does the solution turn blue? What is the composition of the silver coinage of the United States? Write an equation for each of the reactions that take place. When the coin is completely dissolved, evaporate the solution to dryness. The last stages of the evaporation should be carried out cautiously to prevent spattering and consequent loss of material. Now heat the dry residue strongly until it all turns black. What has taken place? What nitrates in general undergo a like thermal decomposition? Boil the black residue for one minute with 15 ml. of water. Filter a *few drops* of the solution through a small filter paper previously wet with water. Test the filtrate with ammonium hydroxide. The appearance of a blue color necessitates evaporation to dryness and further ignition of the residue. Why? If the ammonium hydroxide test for copper gives a negative result, filter off the copper oxide from the 15 ml. of solution containing the silver nitrate. Wash the copper oxide on the filter by pouring through it 20 ml. of water. Combine the last two filtrates and dilute to 100 ml. Each student should take 50 ml. of the silver nitrate solution thus prepared and use it in the following experiments.

180. Reactions of the Silver Ion. *a.* Add a few drops of dilute hydrochloric acid to 5 ml. of silver nitrate solution. Precipitate silver bromide and silver iodide from 5-milliliter portions of silver nitrate solution by the addition of solutions of potassium bromide and potassium iodide. Note the color of each of the silver halides, and list them in the order of their solubility in water. Add 5 ml. of water to each precipitate, and shake until the particles settle readily on standing. Filter, wash once more with 10-milliliter portions of water, and keep the halides for use in *f*.

b. Add a drop of ammonium hydroxide to 2 ml. of silver nitrate solution. What reaction is observed? Now add an excess of the base. Write an equation to express the solution of the brown precipitate. In the same way test the action of sodium hydroxide on silver nitrate solution. Is silver oxide amphoteric? Explain fully the solubility of silver oxide in ammonium hydroxide.

c. Try the effect of sodium carbonate solution on 2 ml. of silver nitrate solution. Write an equation for the reaction. Now heat to boiling. Explain what occurs by writing an equation for the reaction that takes place.

d. Test separately the action of one drop from each of solutions of potassium cyanide and sodium thiosulfate on 2-milliliter portions of silver nitrate solution. Write the equations. Note carefully the changes that take place in the appearance of the precipitated silver thiosulfate. Write the equation. Now try the effect of an excess of potassium cyanide on silver cyanide. Write an equation for the reaction and explain the result.

e. Note the characteristic color of the precipitate formed on the addition of potassium chromate solution to 1 ml. of silver nitrate solution. Test the solubility of the precipitate in dilute nitric acid by the addition of 3 ml. of the acid to the precipitate. Warm gently.

Add a solution of sodium chloride to 3 ml. of silver nitrate solution until no further precipitation takes place. Filter, and add a few drops of a solution of potassium chromate to the filtrate. What is the result? Explain. Look up and record the exact solubility of silver chloride and of silver chromate.

f. Test the action of ammonium hydroxide on small quantities of each of the silver halides prepared in *a.* Note carefully any differences in the behavior of the individual halides, and explain each case in detail. Include equations. Acidify the solution of ammonioargentic chloride with dilute nitric acid. What is the precipitate? What has happened to the ammonioargentic ion?

Determine the solubility of the silver halides in concentrated hydrochloric, nitric, and sulfuric acids.

Try the action of potassium cyanide solution on each of the silver halides. What complex silver ion is formed? Write the equations. Explain the solubility of silver iodide in potassium cyanide solution, and recall your explanation for its failure to dissolve in ammonium hydroxide.

Are the silver halides soluble in sodium thiosulfate solution? Give equations for actions that occur. Refer to your text for the use of sodium thiosulfate in photography. From the standpoint of correct chemical nomenclature show that the use of the term "hypo" is misleading and incorrect.

g. Add a few drops of a solution of any soluble fluoride to 1 ml. of silver nitrate solution. What is the result? Look up and record the exact solubility of silver fluoride.

h. Expose a little silver chloride to direct sunlight. What is the result? Give a brief explanation of the application of this reaction in photography.

i. Pass a few bubbles of hydrogen sulfide through 3 ml. of silver nitrate solution. Is the reaction reversible from a theoretical point of view? Test the validity of your theory. What is the result?

j. Take the remainder of your silver nitrate solution prepared in *a* and add to it dilute hydrochloric acid until precipitation is complete. Filter off the silver chloride and wash it on the filter with 15 ml. of water. Now transfer it to a cavity you have scraped out in a charcoal block, and there mix the halide with approximately equal portions of powdered charcoal and sodium carbonate. Direct the flame from a blowpipe on the mixture, and heat strongly in the reducing portion of the flame until a globule of metallic silver forms. Allow the metal to cool, and then cut it into several pieces. Test the action of dilute hydrochloric and sulfuric acids on the metal. What is the result? What is the action of hot concentrated sulfuric acid on silver? With regard to hydrogen where does silver stand in the activity series of the metals?

181. Properties of Gold. Test the solubility of a small bit of gold leaf in dilute and in concentrated acids. What are the results? Now heat the metal with a mixture of concentrated nitric and hydrochloric acids prepared from 1 vol. of the former and 3 vols. of the latter. What is the common name for this mixture of acids? What compound of gold is obtained when the metal dissolves in the mixture? How is auric chloride obtained from the compound you have prepared? Write the equation. With what valences does gold act?

182. Reactions of Auric Chloride. *a*. Dilute 1 ml. of the stock solution of auric chloride with 10 ml. of water. To this dilute solution add a drop or two of stannous chloride solution. What is the precipitate? Note its color. By what name is it familiarly known? Write an equation to express its formation.

b. Evaporate to dryness in a small porcelain crucible 3 ml. of the stock solution of auric chloride. Now heat the residue gently, taking care that the free flame does not come in contact with the crucible. Note carefully the change that takes place, and write an equation to express the reaction. Continue to heat until the reddish-brown auric chloride has completely disappeared, and then heat strongly. What is the result? What is the residue? Write an equation for the reaction that takes place. What is the general behavior of gold compounds toward heat? Transfer the residue of gold from the crucible to a small cavity in a charcoal block. Heat with a blowpipe until a metallic globule forms. What is the melting-point of gold?

EXERCISES

1. Give equations for the action of concentrated sulfuric and nitric acids on silver.

2. Are all compounds of copper soluble in ammonium hydroxide?

3. How much cupric sulfate is required to make 5 g. of ammoniocupric sulfate?

4. Write the equation for the action of an excess of potassium cyanide on a solution of ammoniocupric sulfate.

5. What is the color of the cupric ion? of anhydrous cupric salts? of cuprous salts?

6. Determine the percentage of copper in brochantite and in tetrahedrite. Which of the copper ores is the most valuable? Does copper occur free in nature?

7. Does silver occur free in nature? Outline briefly the Parkes process for the extraction of silver from lead.

8. What causes silver to tarnish?

9. How are silver mirrors made?

10. Look up the solubility of silver fluoride. How are the other silver halides prepared? Could you prepare silver fluoride by the same method? How should you prepare the compound?

11. How does gold occur chiefly in nature? Can you offer an explanation for your answer?

12. What can you say in general as to the stability of gold compounds toward heat?

CHAPTER XXXV

THE ALKALINE–EARTH METALS

183. The Properties of Calcium. *a.* List the physical properties of calcium.

b. Obtain a small piece of calcium from the storeroom. Cut it in two pieces. Expose one piece to the air for several hours. What change is noted in the appearance of the fresh surface? What compound is formed? Place the other piece on water in a beaker. Note the vigor of the action that takes place. Test the water with litmus paper. Is calcium more or less active with water than is sodium?

c. List the elements with which calcium can combine directly. Write the formulas and names of the compounds which have been formed with each of these elements.

184. Calcium Oxide. *a.* Recall the action of air on metallic calcium. How does the compound formed differ from the one formed by the action of air on sodium?

b. Select two small lumps of marble. Place one lump on a wire gauze and heat it with the full flame of a Bunsen burner for ten minutes. Examine the residue on the gauze and compare its physical properties with those of the unheated marble.

c. Place a little of the quicklime formed in *b* on a piece of red litmus paper and moisten it with one drop of water. What change is noted? Repeat, using a very small fragment of the unheated marble. What change is noted? Explain any difference in the action with litmus of the two substances.

d. Place a small amount of the quicklime produced in *b* in a test tube and add 1 ml. of dilute hydrochloric acid. What change is observed? Repeat this addition of acid, using a very small fragment of marble. What change is observed? Why is there this difference in action between the heated and the unheated substances?

185. Calcium Hydroxide. *a.* Place 2 g. of quicklime (calcium oxide) in an evaporating dish. Add 20 ml. of distilled water. Note the vigor of the action that takes place. When the action has ceased, wash the resultant material into a small flask. Shake the contents of the flask vigorously, allow the suspended matter to settle, and then decant the clear supernatant liquid through a filter paper. Save the filtrate for the following procedure. What is the action of the filtrate on litmus paper?

b. Dilute 1 ml. of the solution from *a* to 2 ml. and exhale your breath through it. Note the precipitate that forms. Continue to blow your breath through the solution until the precipitate dissolves. What caused the precipitate to dissolve? Heat the solution to boiling. Why does the precipitate reappear?

186. Calcium Compounds. *a.* Add a few drops of sodium hydroxide solution to 2 ml. of a solution of calcium chloride. Repeat with a solution of calcium nitrate. What is the precipitate which forms? Repeat these tests, using ammonium hydroxide instead of sodium hydroxide. Explain any differences in action observed. Add one drop of sodium hydroxide solution to 5 ml. of a solution of calcium sulfate. Does a precipitate form? Add several more drops of the sodium hydroxide solution. What result is observed?

b. Add a few drops of a solution of ammonium oxalate to 2 ml. of a solution of calcium chloride. What is the compound which is precipitated? Repeat this test, using a solution of oxalic acid instead of ammonium oxalate. Is any precipitate formed? Add a few drops of ammonium hydroxide solution. Is any change noted? Test the solubility of the precipitate in dilute hydrochloric acid and in dilute acetic acid. Account for any differences noted.

c. Test 2-milliliter portions of a solution of calcium chloride in separate test tubes by adding a few drops of (1) a solution of ammonium carbonate, (2) a solution of ammonium sulfate, (3) a solution of ammonium oxalate, and (4) a solution of sodium chromate. Note the formation or nonformation of precipitates. Now add to each test tube a few drops of ammonium hydroxide. What changes, if any, are noted? Explain the effect of the ammonium hydroxide.

187. Strontium Compounds. *a.* Note the effect of adding a few drops of ammonium hydroxide to 2 ml. of a solution of strontium chloride. Repeat this test, using a few drops of a solution of sodium hydroxide instead of the ammonium hydroxide. Record all observations, and explain any differences noted in the behavior of strontium solutions with the different reagents used.

b. Test 2-milliliter portions of a solution of strontium chloride in separate test tubes by adding a few drops of (1) a solution of ammonium carbonate, (2) a solution of ammonium sulfate, (3) a solution of ammonium oxalate, and (4) a solution of sodium chromate. Does a precipitate form in each case? Add to each test tube a few drops of ammonium hydroxide. Is any change observed? Compare these results with those obtained in the similar experiment with calcium (Experiment 186, *c*).

188. Barium Compounds. *a.* Repeat the tests of Experiment 187, *a*, using a solution of barium chloride instead of strontium chloride. Explain the actions noted. What difference is noted between the solubility of $Ba(OH)_2$ and that of $Sr(OH)_2$?

b. Repeat the tests of Experiment 187, *b*, using a solution of barium chloride instead of a solution of strontium chloride. What differences are noted between the solubilities of the barium salts and the corresponding strontium salts?

EXERCISES

1. State the colors given to the Bunsen flame by the vaporized chlorides of the alkaline-earth metals.

2. Give the formulas of the peroxides of the alkaline-earth metals. What is the product of the reaction of these peroxides with cold dilute acids? Write the equations for such actions.

3. $BaCO_3$ decomposes at a much higher temperature than does $CaCO_3$. From this fact which should you think is the more strongly basic: BaO or CaO?

4. What weight of limestone containing 90 per cent of $CaCO_3$ must be calcined to give 200 tons of quicklime?

5. A dolomitic limestone contains 35 per cent of $MgCO_3$ and 60 per cent of $CaCO_3$. What will be the weight of the lime formed by calcining 10 tons of this stone? What will be the percentage of MgO and of CaO in the lime so formed?

CHAPTER XXXVI

IONIC EQUILIBRIA

189. Repression of Ionization by a Common Ion. *a.* Recall the action of sodium acetate on acetic acid in Experiment 89.

b. Add two drops of 6-*N* ammonium hydroxide to 10 ml. of distilled water. To this solution add four drops of phenolphthalein solution and note the color that is produced. To half the colored solution add an equal volume of a solution of ammonium chloride. What color change is noted? To the other half of the colored solution add an equal volume of distilled water. Compare the results in the two cases. What ion causes phenolphthalein to change from colorless to purplish-red? Does the concentration of this ion increase or decrease upon the addition of ammonium chloride? In what way does this account for your observation? Can you suggest why phenolphthalein was used rather than methyl orange?

190. Displacement of an Acid by Another Acid. Add 1 ml. of a solution of ammonium phosphate to 2 ml. of a solution of ferric chloride. Note the color of the precipitate formed. Divide the contents of the tube into two equal parts. To one add an excess of acetic acid; to the other, an excess of hydrochloric acid. Which acid has the greater solvent action on the precipitate? Look up the ionization of hydrochloric acid, of phosphoric acid, and of acetic acid. From this information explain why hydrochloric acid dissolves ferric phosphate more readily than does acetic acid.

191. Precipitation. Prepare three solutions in separate test tubes as follows: Place in each test tube 5 ml. of distilled water. To the first test tube add one drop of a solution of sodium chloride; to the second, one drop of a solution of potassium chromate; and to the third, one drop of the chloride solution and one drop of the chromate solution. Add silver nitrate solution, drop by drop, to the solution in the first test tube, shaking the tube after each addition until precipitation is complete. What is the color of the precipitate? What is it? In a similar manner note the result of adding a solution of silver nitrate to the contents of the second test tube. What is this substance? Add one drop of silver nitrate solution to the contents of the third test tube. Shake the tube and note the color of the precipitate. What is this precipitate? Add a second drop of silver nitrate solution and shake the tube. What change, if any, is noted? Continue adding the nitrate solution, one drop at a time, until a change in the color of the precipitate is noted. What is the second substance precipitated? Why was it not formed when the first drop of silver nitrate was added? Consult your text, and list the solubilities of each of the substances formed.

EXERCISES

1. Assuming 100-per-cent ionization, calculate the ion-product constants of each of the following substances (obtain molar solubilities from your textbook): (*a*) $BaSO_4$; (*b*) CaC_2O_4; (*c*) $Ba(OH)_2$; (*d*) $MgCO_3$.

2. Assuming complete ionization of the salts, what fraction of a gram of Hg_2Cl_2 would dissolve in 1 liter of 0.1-*N* $AgNO_3$?

3. A mixed solution of chloride and iodide is prepared by adding 10 ml. of 0.1-*N* sodium chloride to 10 ml. of 0.1-*N* sodium iodide. One drop of silver nitrate solution is added to this mixture. What substance will be precipitated? Explain why only one compound is precipitated.

4. Explain clearly why acetic acid will dissolve $BaCO_3$ and will not dissolve $BaCrO_4$.

5. Why will NH_4Cl prevent the precipitation of $Mg(OH)_2$ by NH_4OH?

CHAPTER XXXVII

MAGNESIUM, ZINC, CADMIUM, AND MERCURY

192. Properties of Magnesium. *a.* Grasp a piece of magnesium ribbon 5 cm. long with the crucible tongs and hold it in the flame of the Bunsen burner. What takes place? Would a copper strip suffer a similar change if placed in the flame?

b. Place 0.5 g. of magnesium ribbon or turnings in a crucible provided with a tightly fitted cover. Support the crucible on a pipestem triangle, and heat it and the contents for ten minutes with as hot a Bunsen flame as it is possible for you to obtain. Lift the crucible cover from time to time. Allow the crucible to cool, and examine the residue. Can you discern a yellow substance mixed with the white magnesium oxide? Transfer the contents of the crucible to a test tube and add a few milliliters of distilled water. Note the development of heat. Test the effect of the liquid in the tube on litmus paper. Test the escaping gas at the mouth of the tube with moist litmus paper. What causes the observed action? Note the odor of the escaping gas. What is the compound being given off? How was it formed?

c. Test the action of dilute sulfuric acid on small pieces of magnesium ribbon. Repeat with dilute nitric acid and with sodium hydroxide. Identify any gases which may be given off.

193. Magnesium Oxide. *a.* Place a fragment of magnesium carbonate (magnesite) on a wire gauze and heat it strongly with the flame of a Bunsen burner. Note the physical properties of the product. Place a little of this material on a piece of red litmus and moisten the powder with a drop of water. Why does the litmus change in color?

b. Make a paste of magnesium oxide and water, and heat it on charcoal with the flame of a blowpipe. Does the oxide fuse? Allow the heated material to cool, and moisten it with a drop of a dilute solution of cobalt nitrate. Again heat the material and note the color produced.

194. Magnesium Compounds. *a.* Add several drops of sodium hydroxide to 1 ml. of a solution of magnesium chloride. What is the precipitate which forms? Repeat with another 1-milliliter portion of the solution of magnesium chloride, substituting ammonium hydroxide for sodium hydroxide. Does a precipitate form? Test a third 1-milliliter portion of magnesium chloride solution, using, first, several drops of a solution of ammonium chloride, and then a few drops of ammonium hydroxide. Does a precipitate form? Add several drops of sodium hydroxide to the contents of the third test tube. Does a precipitate form? Account for the differences in behavior noted.

b. Add to 3-milliliter portions of a solution of magnesium chloride in separate test tubes a few drops of (1) a solution of ammonium carbonate, (2) a solution of sodium carbonate, (3) a solution of ammonium hydrogen phosphate, and (4) a solution of ammonium sulfate. Note the formation of any precipitates. Then add several drops of ammonium hydroxide to each solution. Note any changes which may take place. Compare the solubilities of magnesium salts with those of the corresponding calcium salts.

195. Properties of Zinc. *a.* Hold a piece of zinc in the flame of the Bunsen burner. Does zinc burn readily? How could it be burned? Compare it with magnesium in this respect.

b. Place 0.5 g. of granular zinc in a test tube and add to it 5 ml. of dilute hydrochloric acid. Test the escaping gas. What is it?

c. Add 5 ml. of sodium hydroxide to 0.5 g. of granular zinc. What is the gas that is formed?

d. Add 5 ml. of 3-*N* nitric acid to 0.2 g. of granular zinc. Note the character of any gases which may be evolved. When the action in the tube has ceased, cool the solution, and add sodium hydroxide to it until it is alkaline. Warm the tube and contents gently, and note the odor of the escaping gas. Test it with moist red litmus. What is the compound formed? Explain the formation.

196. Zinc Compounds. *a.* Place a very small amount of zinc nitrate on a piece of charcoal and heat it with the flame of a blowpipe. Allow the material to cool, and moisten it with a drop of a dilute solution of cobalt nitrate. Heat it again with the flame of a blowpipe and note the color produced.

b. Place 2 ml. of a solution of zinc chloride in a test tube and add a few drops of dilute ammonium hydroxide. Note the white gelatinous precipitate which forms. Add more ammonium hydroxide until the precipitate dissolves. Account for the dissolving of the precipitate.

c. Repeat *b,* using a solution of sodium hydroxide instead of ammonium hydroxide. Does the white precipitate appear? Explain the observed changes.

d. Add dilute ammonium hydroxide to a solution of zinc chloride until the initial precipitate dissolves. Add dilute acetic acid until the solution will turn blue litmus red. Saturate this solution with hydrogen sulfide. What is the white precipitate which forms? Add dilute hydrochloric acid to the contents of the tube. Why does the precipitate dissolve in hydrochloric acid but not in acetic acid?

197. Cadmium Compounds. *a.* Add a few drops of ammonium hydroxide to 2 ml. of a solution of cadmium chloride. What is the precipitate that forms? Continue adding ammonium hydroxide, drop by drop, until the precipitate dissolves. Account for this action. Repeat this test, using a solution of sodium hydroxide? Do you note any differences in the action? Explain your observations. Compare your results with those obtained in the experiments on zinc.

b. Saturate 2 ml. of a solution of cadmium chloride with hydrogen sulfide. What is the color of the precipitate? Test its solubility in hydrochloric acid. Explain your observations.

198. Properties of Mercury. *a.* Place several drops of mercury in a porcelain dish. Examine the metal carefully and describe its physical properties. Shake the dish carefully. What takes place?

b. Pour one drop of the mercury into a hard-glass test tube. Heat the tube and contents gently. Is there any deposit formed on the cool upper part of the tube? If so, examine it carefully and decide what it is. What property of mercury is illustrated by this experiment?

c. Place a copper cent in an evaporating dish. Clean the coin by adding 5 ml. of dilute nitric acid and allowing the coin to remain in the liquid for one minute after the action has started. Remove the coin, wash it once with water, and place it in the dish containing mercury. Push the coin about until all parts of it have come in contact with the mercury. Remove the coin and rub it with a cloth. What caused the change in its appearance?

d. Place 15 ml. of a solution of silver nitrate in a small beaker. Add two drops of mercury. Note the treelike growth of silver amalgam (Diana's garden).

199. Mercurous Compounds. *a.* Add two drops of hydrochloric acid to 2 ml. of solution of mercurous nitrate. What is the precipitate that forms? What is its formula (consult your textbook)?

b. Add an excess of ammonium hydroxide to the precipitate obtained in *a.* What are the substances that are formed?

c. Add two drops of potassium iodide solution to 2 ml. of a solution of mercurous nitrate. What substance is formed?

200. Mercuric Compounds. *a.* Add two drops of hydrochloric acid to 2 ml. of mercuric nitrate solution. Compare the result with that obtained in Experiment 199, *a.*

b. Add ammonium hydroxide, drop by drop, until a decided action has been manifested. What is the compound that is formed? Compare this action with that in Experiment 199, *b.*

c. Add two drops of stannous chloride solution to 2 ml. of a solution of mercuric chloride. Note the color of the precipitate that is formed. What is this substance? Add an excess of stannous chloride. What change is noted? Explain this action.

d. Add two drops of a solution of potassium iodide to 2 ml. of a solution of mercuric chloride. Shake the tube to coagulate the precipitate. What is the color of the precipitate? Does it change color on standing? Filter out this substance and wash it with water. Dry it between filter papers and transfer the powder to a hard-glass test tube. Heat the tube and contents gently. Is there any change in color? Allow the test tube to cool, and note any changes which take place. Scratch the material with a glass rod. What effect is noted? Add 2 ml. of a solution of potassium iodide to the material in the test tube. Does the compound dissolve in this solution? Explain.

EXERCISES

1. What are the usual ores of zinc? of cadmium?

2. What is *hydrolysis*? With what types of salts is it most complete?

3. Is the action of magnesium nitride with water to be considered as hydrolysis? If so, state which of the products is an acid and which is a base.

4. What are the commonest rocks containing magnesium?

5. What are the usual ores of mercury?

6. What is *ammonolysis*?

7. Does mercuric nitrate resemble mercuric chloride in its action with ammonia?

CHAPTER XXXVIII

ALUMINUM AND THE METALS OF GROUP III

201. Properties of Aluminum. *a.* Examine a piece of aluminum wire and record its physical properties, such as color, hardness, and ductility. Look up the density of the metal. Try to burn the wire in the Bunsen flame. Now drop a pinch of aluminum powder into the flame. What is the result? What is formed? Write an equation for the reaction. Why were you unable to make the wire burn?

b. Place a few aluminum turnings in each of three test tubes, and try the action of the metal with dilute hydrochloric acid, with dilute nitric acid, and with dilute sulfuric acid. Warm the solutions, if necessary, to start the action. With which acid does the metal act most readily? Refer to your text, and give an explanation for the difference in activity of the metal toward hydrochloric acid and toward the two oxidizing acids.

c. Test the action of sodium hydroxide solution on aluminum turnings. If a reaction does not take place in the cold, try the effect of heat. What gas is evolved? Write an equation for the reaction.

202. Reactions of Aluminum Salts. *a.* Add a solution of sodium hydroxide, drop by drop, to 5 ml. of a solution of an aluminum salt until no more precipitate is formed. Write an equation for the reaction. Note the color and gelatinous character of aluminum hydroxide. Now add an excess of sodium hydroxide solution. Shake the test tube continuously during the addition of the base. What is the result? Write the equation.

b. Repeat *a*, using ammonium hydroxide in place of sodium hydroxide. Difference in result?

c. Precipitate aluminum hydroxide from 5 ml. of a solution of aluminum chloride by the use of ammonium hydroxide. Filter off the precipitate, wash it with distilled water, and then transfer it to a small watch glass. Now add dilute hydrochloric acid to the hydroxide. What is the result? Write the equation for the reaction.

How does aluminum hydroxide react in the presence of an acid? in the presence of a base? How do you classify a hydroxide of this type? What could you say about the strength of aluminum hydroxide as a base? as an acid? What can you predict concerning the degree of hydrolysis of aluminum salts? Give a possible explanation for the failure of aluminum hydroxide to dissolve in ammonium hydroxide.

d. Test separately, with litmus paper, solutions of aluminum chloride, sulfate, and nitrate. Explain the results obtained.

e. Treat 5 ml. of a solution of aluminum chloride with 5 ml. of colorless ammonium sulfide. Filter off the precipitate and wash it with water until the odor of hydrogen sulfide is no longer perceptible. Write the equation for the double-decomposition reaction that would be expected to take place. How should you ascertain whether or not the precipitate is aluminum sulfide? Test out your procedure and record the result. What is the precipitate? Write equations showing the reactions that do take place on the addition of a sulfide to a solution of an aluminum salt. Why is the hydrolysis of aluminum sulfide complete? Show that this experiment verifies your prediction regarding the strength of aluminum hydroxide as a base. How could aluminum sulfide be prepared?

f. Add a solution of sodium carbonate to 5 ml. of a solution of aluminum chloride. Determine whether or not the precipitate is aluminum carbonate. Write equations for the reactions that have taken place.

203. Preparation of "Ordinary" Alum. Dissolve 0.02 of a gram-molecular weight of hydrated aluminum sulfate, $Al_2(SO_4)_3 \cdot 18\,H_2O$, in 25 ml. of water. Now heat the solution to boiling and add the calculated amount of finely powdered potassium sulfate. Keep the solution at the boiling-point and shake it until all the potassium sulfate has dissolved. Filter the hot solution into a small beaker, cover the beaker with a watch glass, and set it aside in your desk until the next laboratory period. Usually a single large mass composed of smaller crystals will be formed. Remove the alum and dry it by pressing it between sheets of filter paper. Touch your tongue to the mass and note the taste. Refer to your text for the form of the crystals. Are the crystals of all alums isomorphous? What is the general formula of an alum? Name the other elements of Group III that form alums. Does a solution of an alum contain a complex ion? To what class of salts do the alums belong? Can you precipitate aluminum hydroxide from a solution of the alum you have prepared?

204. Test for Aluminum. *a.* Add a drop of a solution of cobalt nitrate to 3 ml. of the aluminum sulfate solution on the reagent shelf. To this mixture add an excess of ammonium hydroxide, filter off the precipitate that is formed, and wash it with water. Make as small a loop as possible at the end of a platinum wire, and collect in this loop some of the precipitate on the filter paper. Now carefully moisten this lump of precipitate with a very small amount of concentrated sulfuric acid (the acid should be applied with a clean thin glass rod which has been previously thrust into a test tube of the acid). Now heat the material on the wire in a Bunsen flame until a dull dark-blue mass of cobalt metaluminate (Thénard's blue) is formed. Write an equation for this reaction between the oxides of cobalt and aluminum. Some dexterity is required to prevent the precipitate, which shrinks on ignition, from falling off the wire. If silica is present, a shiny fusible blue bead is formed in the loop of the wire. This is readily distinguishable from the dull-blue mass of cobalt metaluminate which will coat the wire above the loop.

b. Obtain a precipitate of aluminum hydroxide by adding ammonium hydroxide to 3 ml. of a solution of aluminum chloride. Filter off the precipitate, wash it once with water, and then dissolve it by pouring 5 ml. of normal hydrochloric acid through the filter. Treat the solution with 8 ml. of 2-*N* ammonium acetate and 3 ml. of a 0.5-per-cent solution of the ammonium salt of aurin tricarboxylic acid. Shake the tube thoroughly and allow it to stand. Within a few minutes a bright-red insoluble aluminum salt of the aurin tricarboxylic acid will be formed. If the ions of the alkaline-earth metals are present in high concentrations, red precipitates will also be formed. However, if a soluble carbonate is added to the precipitates formed by these metals, the more insoluble alkaline-earth carbonates will be formed, thus causing the disappearance of the red aurin tricarboxylates. Therefore, to confirm the presence of aluminum, it is necessary to render the solution above the red precipitate alkaline with ammonium hydroxide, to add several milliliters of ammonium carbonate solution, and to note whether or not the red precipitate persists.

205. Formation of Lakes. *a.* Add 0.1 g. of eosin (*storeroom*) to 5 ml. of water which has been made ammoniacal with three drops of ammonium hydroxide. Now add 3 ml. of aluminum sulfate solution, shake, and heat to boiling. Filter off the precipitate that has formed and wash it thoroughly with water. Note the color of the precipitate. When a dye combines with or is absorbed by a hydroxide such as aluminum hydroxide or chromium hydroxide, the resulting colored precipitate is known as a "lake."

b. Make 5 ml. of cochineal solution ammoniacal with a few drops of ammonium hydroxide and then add 2 ml. of aluminum sulfate solution. Shake the mixture, filter off the lake, and note its color.

206. Aluminum Hydroxide as a Mordant. Place a piece of cotton cloth in a test tube containing a solution of aluminum sulfate prepared by diluting 3 ml. of the shelf reagent with 10 ml. of water. Boil the liquid three minutes. In another test tube dilute 1 ml. of ammonium hydroxide with 10 ml. of water, and immerse the cloth in this solution. Bring the latter just to the boiling-point and then shake it for two minutes. The cloth is now said to be "mordanted" with aluminum hydroxide.

Obtain from the storeroom 10 ml. of an aqueous suspension of alizarin and mix it well with 50 ml. of water contained in a beaker. Immerse the piece of mordanted cloth in this suspension, together with a piece which has not been so treated. Heat the mixture just to boiling and stir the pieces of cloth with a glass rod for ten minutes. At the end of this time remove the cloths and wash them in running water from the tap. On which piece of cloth is the dye fast? Account for the difference.

EXERCISES

1. How was the element indium discovered?

2. With what element is indium associated in its ores?

3. What are the valences of indium?

4. Are the hydroxides of indium amphoteric?

5. How was thallium discovered? Tell why it received the name *thallium.*

6. Write the formulas of the hydroxides of thallium. Are they amphoteric?

7. In what division of Group III are scandium and yttrium placed? With what valence do they act?

8. Why are the rare-earth elements assigned to a single space in the periodic table? Why are they so difficult to separate from one another?

9. Do any rare-earth elements remain undiscovered? What is the most recent one to be discovered? Where, and by whom, was it discovered?

10. Do the rare-earth elements or their compounds have any commercial value?

11. What is the usual valence of the rare-earth elements? Do they form alums?

CHAPTER XXXIX

COLLOIDS

207. Emulsions. Obtain from the storeroom about 25 ml. of kerosene or similar oil. Also obtain about 50 to 60 ml. of a 1-per-cent soap solution.

Shake together about 5 ml. of oil with an equal amount of water. An emulsion will form which is not stable. Now place about 20 ml. of oil in a 200-milliliter Erlenmeyer flask and add to the oil about 50 ml. of the soap solution. Shake well and let stand for half an hour. A rather stable emulsion should form. This is probably due to the lowering of the surface tension of water. The water is all "bound" to the oil droplets. A cream will rise on this emulsion. With a glass tube or pipette draw off a part of this "cream" and place it in a test tube. In a second test tube place an equal amount of the emulsion below the "cream."

To each of these test tubes add two or three milliliters of dilute hydrochloric acid. This will cause the emulsion to break. Observe the oil layer in each tube. Did the "cream" have a greater or a less percentage of oil?

208. Peptization of Precipitates. Place in a flask about 15 to 20 ml. of a solution of a cadmium salt ($CdSO_4$). Add enough ammonium sulfide to precipitate the cadmium sulfide completely. Wash it with distilled water. Remove the precipitate from the filter paper and place it in an Erlenmeyer flask. Add distilled water and shake the mixture to suspend the precipitate in the water thoroughly. Now pass in hydrogen sulfide until the precipitate has become completely dispersed in the solution. The excess hydrogen sulfide may be boiled off from this colloidal solution of cadmium sulfide. The sulfide ion acts as the peptizing agent in this case.

209. Preparation of Colloidal Silver by Reduction with Tannin. Obtain from the stockroom 5 ml. of a 1-per-cent silver nitrate solution. Add very dilute ammonium hydroxide, drop by drop, until the precipitate which forms just disappears. Then add about 90 ml. of distilled water. Next add one or two milliliters of a tannin solution (0.3 to 0.4 g. of tannin per 100 milliliters of water). Repeat, using hot solutions. The colloid should be olive green by transmitted light.

210. Preparation of Colloidal Ferric Hydroxide. Obtain from the storeroom 5 ml. of concentrated ferric chloride solution. Heat 300 ml. of distilled water until it boils and then pour the ferric chloride into the boiling water. The stable red solution which forms (by hydrolysis) is a colloidal solution of ferric hydroxide.

211. Diffusion (the "Patriotic Tube"). Dissolve 1 g. of agar-agar in 40 ml. of warm water. Place this solution in a large test tube. Add to the solution several drops of phenolphthalein. If the solution becomes pink, add acetic acid to it, drop by drop, until the liquid becomes colorless. Add to the solution enough sodium hydroxide to bring back the pink color. Treat the resulting solution with 0.5 ml. of a solution of potassium ferrocyanide and set the mixture aside until it sets to a solid jelly. After the setting has taken place, pour 0.5 ml. of a solution of ferric chloride on top of the agar and allow the tube to stand. As the ferric ions diffuse they form, with the ferrocyanide ions, an advancing blue band; the hydrogen ions, which diffuse more rapidly, discharge the pink color ahead of the blue band. In about two days the tube will have bands of red, white, and blue.

212. Imitation Fruit Jelly. Dissolve 0.5 g. of pectin in 50 ml. of distilled water. Add to this solution 0.25 g. of tartaric acid. When the acid has dissolved, heat the liquid to boiling and add to it 30 g. of cane sugar. Concentrate the solution by boiling it. When the temperature of the solution reaches 105°, set the mixture aside to cool. A firm jelly should be obtained.

CHAPTER XL

THE ELEMENTS OF GROUP IV

213. Properties of Tin. *a.* Place a small piece of tin on a piece of charcoal and heat it in the oxidizing flame of the blowpipe. Note the color of the coating which forms on the tin. Does the color change when the substance is allowed to cool to room temperature?

b. Place 0.5 g. of tin in a test tube and add to it 5 ml. of concentrated hydrochloric acid. Heat the mixture to boiling (*CARE!*). Test the gas which is evolved by applying to it a lighted splint. Dilute the residual liquid to 20 ml. and saturate the solution with hydrogen sulfide.

c. Place 0.5 g. of tin in a test tube and add to it 5 ml. of concentrated sulfuric acid. Heat the mixture to boiling (*CARE!*). Note cautiously the odor of the gas which is formed. Allow the solution to cool and pour it into 20 ml. of distilled water. Filter off any precipitate. Saturate the filtrate with hydrogen sulfide.

d. Treat 0.5 g. of tin with 5 ml. of concentrated nitric acid. If no action takes place, add one or two drops of water. Note the color of the gas which is formed. Divide the white solid so formed into two parts. To the first add 10 ml. of sodium hydrogen sulfide solution. Make the resulting solution just acid by adding dilute hydrochloric acid. What color is the precipitate so formed? Dissolve the second portion of the white solid in concentrated hydrochloric acid. Dilute this solution to five times its original volume and saturate it with hydrogen sulfide. What is formed?

e. Place 0.5 g. of tin in a small evaporating dish and add to it 10 ml. of sodium hydroxide solution. Bring the solution to the boiling-point. Add more sodium hydroxide, if necessary, to bring about complete solution of the tin. When all the tin has dissolved, transfer the solution to a flask and add dilute hydrochloric acid until the solution is just acid to litmus. Then saturate the solution with hydrogen sulfide. What is formed?

214. Stannous Compounds. *a.* Recall the actions with hydrogen sulfide of the solutions formed in Experiments 213, *b*, and *c*. What color is stannous sulfide?

b. Place 1 ml. of mercuric chloride solution in a test tube and add to it one drop of stannous chloride solution. If no precipitate forms, add a second drop of stannous chloride. Filter off this precipitate and moisten it on the filter with ammonium hydroxide solution. What class of mercury compounds is indicated?

c. Repeat the initial part of *b*, but now add an equal volume of stannous chloride solution. If any white precipitate persists, add an excess of stannous chloride. How do you explain the change in color of the precipitate?

d. Place 2 ml. of a solution of stannous chloride in a test tube and add to it a few drops of dilute sodium hydroxide solution. Filter out the precipitate which forms. Divide this solid into two portions. Heat the first portion gently in a crucible. Note the color of the product so formed. Dissolve the second portion in excess sodium hydroxide. Test the reducing power of this solution by adding a few drops of it to some freshly precipitated bismuth hydroxide.

215. Stannic Compounds. *a.* Recall the action with hydrogen sulfide of the solution formed in Experiments 213, *d*, and *e*. What is the color of stannic sulfide?

b. Test the action of stannic chloride with mercuric chloride. Add an iron nail and 2 ml. of hydrochloric acid to 5 ml. of stannic chloride solution. When the evolution of gas has ceased, test the resultant solution by adding to it a few drops of mercuric chloride. Explain the results you obtain.

c. Place 5 ml. of a sodium stannate solution in a flask. Add dilute hydrochloric acid until the solution is just acid to litmus. Then saturate the solution with hydrogen sulfide. Filter out the precipitate and dissolve it in sodium hydrogen sulfide solution. Make the resultant solution just acid with dilute hydrochloric acid. What is the compound which is precipitated?

216. Properties of Lead. *a.* Note the physical properties of metallic lead. Can it be cut with a knife?

b. Place a very small piece of lead in a cavity made in a stick of charcoal. Heat the lead with the oxidizing flame of a blowpipe. Note the color of the incrustation formed on the sides of the cavity. What is this incrustation? Does it change color when it is allowed to cool to the temperature of the room?

c. Place 0.5 g. of lead in an evaporating dish and add to it 20 ml. of water and 5 ml. of concentrated nitric acid. Warm the mixture gently until the lead is dissolved. Test a 5-milliliter portion of the resultant solution with dilute hydrochloric acid. Test a second 5-milliliter portion with potassium iodide solution and a third portion of the same volume with sulfuric acid. What is the precipitate in each case? Note the color and general appearance of each precipitate.

217. Oxides of Lead. *a.* Write the names and formulas of the various oxides of lead.

b. Dissolve 0.5 g. of lead monoxide in an excess of nitric acid. Add to 1 ml. of this solution 1 ml. of potassium iodide solution. Reserve the remainder for *c.*

c. Add dilute sodium hydroxide, drop by drop, to the solution *b.* When the solution becomes basic, filter out the precipitate formed. Dissolve half the precipitate in dilute hydrochloric acid and the other half in a sodium hydroxide solution. Why does this precipitate act in this way?

d. Add sodium hydroxide solution to 4 ml. of lead nitrate solution until the precipitate which forms at first is redissolved. Then add strong chlorine water, or saturate the solution with chlorine from a chlorine tank or generator. Note the dark-brown precipitate which forms. Filter out this precipitate and wash it several times with water. Transfer the dark-brown substance to a test tube and shake it up with 8 ml. of water. Divide the suspension equally among four test tubes. Add to the respective tubes (1) dilute nitric acid, (2) dilute sulfuric acid, (3) dilute hydrochloric acid, and (4) sodium hydroxide solution. Record your observations. Explain.

e. Place 0.5 g. of red lead in a hard-glass test tube and ignite it in the Bunsen flame. Test the escaping gas with a glowing splint. What change takes place in the test tube?

f. Add 10 ml. of concentrated nitric acid to 2 g. of red lead in an evaporating dish. Warm gently, if necessary, to start the action. Dilute the solution to double its volume, and filter out the residue. Does it resemble the original red lead? Wash the residue well with water. Test the filtrate with potassium iodide solution. Treat the residue with hydrochloric acid. Discuss your results.

218. Plumbous Compounds. *a*. Recall the action of plumbous salts with chlorides and iodides.

b. Add a few drops of dilute hydrochloric acid to 1 ml. of lead nitrate solution. Filter out the precipitate and wash it repeatedly with boiling water. What is noted about the solubility of lead chloride?

c. Saturate 2 ml. of lead acetate solution with hydrogen sulfide. What color is lead sulfide?

d. Add several drops of lead acetate solution to 1 ml. of potassium chromate solution. Is the precipitate which forms soluble in acids? in strong bases?

e. Place 5 ml. of lead acetate solution in a small beaker and add sodium carbonate solution, drop by drop. Is there any effervescence? Filter out the precipitate which forms and wash it with water. Spread it out between filter papers and allow it to dry in the air. When dry, place some of the white powder in a hard-glass test tube and heat it. What is the yellow powder which results?

EXERCISES

1. What are the common ores of lead and of tin?

2. Arrange a table of the dioxides of the elements of Group IV, giving the names and formulas.

3. Do the compounds of any other elements of Group IV resemble those of silicon?

4. State which is the more acidic: stannous oxide or stannic oxide. Give reasons for your answer.

5. Give evidence to show whether stannous oxide or lead monoxide is more basic.

6. Is lead monoxide an acidic oxide or a basic oxide?

7. Distinguish between *litharge* and *massicot*.

CHAPTER XLI

CHEMISTRY IN LIVING PROCESSES

219. Photosynthesis. Arrange the apparatus shown in Fig. 59. Place fresh green leaves in the 300-milliliter flask, which is then filled with water previously saturated with carbon dioxide. The stopper is pushed in, and the funnel is filled with water charged with carbon dioxide. Fill the test tube with the same liquid and arrange it as shown in the figure. Allow the apparatus to stand in the sunlight for several hours. What is the gas which collects in the test tube? What is its origin? Test the gas with a glowing splint.

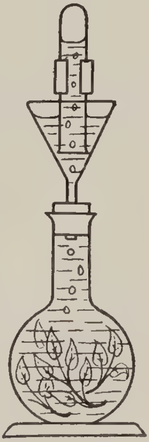

FIG. 59

220. Acid or Alkaline Soils. Place a red and a blue piece of litmus paper on separate watch glasses. Make a paste with 0.5 g. of the soil to be tested and a small amount of distilled water. Place a little of this paste on each piece of litmus. After fifteen minutes note any change in the colors of the litmus.

221. Measurement of Acidity of Soils. Place 50 g. of an acid soil in a wide-mouthed bottle. Add to the soil 125 ml. of a 0.1-*N* potassium nitrate solution. Stopper the bottle tightly and shake it vigorously at five-minute intervals for an hour. Allow the mixture to stand until the next laboratory period. Pipette off 25 ml. of the clear supernatant liquid, boil it for ten minutes to expel all the carbon dioxide, and titrate it with a standard (1-*N*) solution of sodium hydroxide. Use phenolphthalein as an indicator. Repeat the titration with another 25-milliliter portion of the clear liquid. From the average of your determinations calculate the amount of calcium carbonate that would be needed to counteract the acidity of the soil used.

CHAPTER XLII

CHROMIUM AND MANGANESE

222. Preparation of Chrome Alum. Dissolve 5 g. of potassium dichromate in 20 ml. of water and add to the solution the calculated amount of sulfuric acid necessary to form potassium sulfate and chromic sulfate. Warm the mixture and add 10 ml. of alcohol, a few drops at a time (*CARE!*), until a dark-green solution results (this action may take some time for completion). Note the odor given off. Carefully add 10 ml. of a solution of potassium hydroxide. Evaporate the solution to about half its original volume and allow it to cool. Describe any crystals which separate.

223. Reactions of the Chromic Ion. *a.* Add ammonium hydroxide, drop by drop, to 2 ml. of a solution of chrome alum. Describe the precipitate which forms. Save this precipitate for a following experiment.

b. Add sodium hydroxide, drop by drop, to 2 ml. of a solution of chrome alum. When the precipitate which forms at first has completely dissolved, boil the solution for a moment. Will the precipitate which forms dissolve in excess sodium hydroxide?

c. Add 1 ml. of a solution of colorless ammonium sulfide to 2 ml. of a solution of chrome alum. Filter off the precipitate which forms and wash it well with water. Is this precipitate a sulfide? How can you test it to find out? If your instructor approves your proposed test, try it out. What is the precipitate?

224. Chromates and Dichromates. *a.* Treat the precipitate from Experiment 223, *a*, with enough sodium hydroxide to dissolve it. Stir hydrogen peroxide into the resulting solution until the liquid assumes a bright yellow color. To what ion is the color due? Add 1 ml. of a solution of sodium carbonate to the solution and boil it until it is about half its original volume. By this treatment the excess peroxide is decomposed.

b. To the solution from *a* add sulfuric acid, drop by drop, until the solution is distinctly acid to litmus. What causes the change in color? Write an ionic equation expressing the chemical action.

c. To the solution from *b* add sodium hydroxide until the mixture is alkaline to litmus. What color change is noted? Write an ionic equation for the chemical action.

225. Sparingly Soluble Chromates. Treat separate 2-milliliter portions of a solution of potassium chromate with an equal volume of a solution of each of the following: silver nitrate, barium chloride, lead acetate, and zinc sulfate. Give equations for the formation of these precipitates. Note their colors.

226. Test for Chromates or Dichromates. Acidify 5 ml. of water containing three or four drops of potassium dichromate with two or three drops of dilute sulfuric acid. To the solution add 5 ml. of ether and shake the mixture; then add a drop or two of hydrogen peroxide and shake out the solution with ether. The ether dissolves out the blue color. How could this test be applied to the detection of chromium in a lower state of oxidation? An excess of either dichromate or hydrogen peroxide renders the test less delicate or may destroy the blue color entirely.

227. Properties of the Manganous Ion. *a.* To 2 ml. of a solution of manganous sulfate add an equal volume of a solution of ammonium chloride. Make the solution alkaline with ammonium hydroxide. Expose the mixture to the air and note any change in it. Explain the results.

b. Add sodium hydroxide to 2 ml. of a solution of manganous sulfate until the precipitation is complete. Filter, and treat the precipitate first with 2 ml. of water and then with a small amount of sodium peroxide. Remove the solid by filtration and fuse it on a piece of charcoal with sodium carbonate and sodium chlorate. What is the color of the product? What compound produces this color? Remove the fused mass to a test tube about one third full of water. Pass chlorine into the solution and note the color of the resulting liquid. What ion produces the color?

228. Reduction of Permanganates. Place 5 ml. of a neutral solution of potassium permanganate in a 50-milliliter Erlenmeyer flask. Saturate the solution with hydrogen sulfide. Note the precipitate which forms. What is it? Now add sulfuric acid until the solution is acid to litmus, and again saturate with hydrogen sulfide. What color is the solution after this action? What is the solid material left in the liquid? How may its identity be established?

229. Test for the Manganous Ion. Boil 2 ml. of a solution of manganous sulfate with 0.5 g. of lead dioxide and 5 ml. of concentrated nitric acid. After the mixture has been boiled for one minute or more, allow it to stand until any solid settles to the bottom of the container. Note the color of the supernatant liquid. What acids are present in this solution? Which one produces the color? If manganous chloride were used, would hydrochloric acid be present in solution at the end of the reaction?

EXERCISES

1. State the color of the following ions: (1) manganous, (2) manganic, (3) permanganate, (4) manganate, (5) chromous, (6) chromic, (7) chromate, (8) dichromate.

2. What recently discovered elements complete the manganese family?

3. Which oxide of vanadium should you expect to be the most acidic?

4. For what are vanadium and columbium used?

5. Can you suggest why tungsten is chosen instead of other metals for making filaments for electric lights?

6. What kind of salts does uranium usually form?

7. What are the chief uses of molybdenum?

CHAPTER XLIII

IRON, COBALT, AND NICKEL

230. Properties of Iron. *a.* Clean a three-inch piece of iron wire by scraping the surface with the blade of a knife. Heat the wire for a minute in the hottest part of the Bunsen flame. Is the metal a good conductor of heat? Remove the wire and note any change in its appearance. What is the coating? Recall the product formed by the burning of iron in oxygen. Drop a pinch of powdered iron into the Bunsen flame. What result is observed? What is the product? What is the action of steam on red-hot iron? What is formed by the slow oxidation of iron in the presence of moisture?

b. Weigh accurately a small triangular file and then immerse it completely in a known volume of water contained in a graduated cylinder. Note the increase in the volume of the water, and then calculate the specific gravity of the steel.

c. Heat a short piece of steel wire to redness and then drop it immediately into cold water. Now try to bend the wire. What is the result? Use the luminous flame of the burner to reheat the wire, and then allow it to cool in the air. Can the wire be bent now? Refer to your text for a discussion of the heat treatment of steel.

d. Test the action of iron with dilute hydrochloric, sulfuric, and nitric acids. What results are observed? Write equations for all the reactions taking place. Recall the action of fuming nitric acid on iron (Experiment 116, *d*). Determine the position of iron in the activity series of the metals by placing short pieces of *clean* iron wire in solutions containing ions of magnesium, aluminum, zinc, cadmium, nickel, lead, copper, mercury, and silver. Test separately the action of zinc and of lead on ferrous sulfate solution. Write equations for the actions that take place.

231. Tests for the Ferrous and Ferric Ions. *a.* To 15 ml. of dilute sulfuric acid contained in a test tube add several small iron nails. Heat the acid to the boiling-point. Set the tube aside and allow the reaction to continue for fifteen minutes. Use this solution of ferrous sulfate in the tests for ferrous ion, Fe^{++}. Add a few milliliters of ammonium hydroxide to 2 ml. of ferrous sulfate solution. Quickly note the color of the precipitate and then shake the solution vigorously. What is the result? What was the original precipitate?

b. Add ammonium hydroxide to 3 ml. of ferric chloride solution. Note the color of the precipitate. What is the compound? Compare it with the substance obtained by shaking the precipitate of ferrous hydroxide with air. What is the effect of air on ferrous hydroxide? Write equations for all reactions taking place.

c. Test the action of a solution of potassium ferrocyanide with solutions of ferrous sulfate and ferric chloride. What results are observed? Write equations for the reactions. What is the common name for ferric ferrocyanide?

d. Treat solutions of ferrous sulfate and ferric chloride with a solution of potassium ferricyanide. What results are observed? What is the common name for the precipitate obtained with ferrous sulfate? Write an equation to express its formation. How can the ferrous ion be detected in the presence of the ferric ion?

e. Add one drop of a solution of ammonium thiocyanate to 2 ml. of ferrous sulfate solution. What is the result? Test ferric chloride solution with a drop of a solution of ammonium thiocyanate. What is the soluble colored substance? Write the equation. The formation of this compound constitutes a very delicate test for the ferric ion.

f. Dilute one drop of ferric chloride solution with 50 ml. of water. Test 5 ml. of this solution with ammonium thiocyanate solution. What is the result? Now add 2 ml. of ether to this faintly colored solution, stopper the test tube with your thumb, and shake vigorously. Allow the ether to separate. What is present in the ether layer? What can you say as to the relative solubility of ferric thiocyanate in water and in ether? How does the extraction of the ferric thiocyanate in ether increase the delicacy of this test for the ferric ion? Test the tap water for ferric iron.

g. Record in tabular form the results obtained in *a, b, c, d, e,* and *f*.

232. Oxidation of Ferrous Salts. *a*. Dissolve 1 g. of ferrous sulfate in 10 ml. of water. Reserve 5 ml. of this solution for use in *b*. To the remainder add a few drops of dilute sulfuric acid and 5 ml. of dilute nitric acid. Boil the mixture for one minute. Test separate portions of the product for ferrous and ferric ions. What are the results? Write an equation for the oxidation of the ferrous salt.

b. To 5 ml. of the ferrous sulfate solution prepared in *a* add 1 ml. of concentrated hydrochloric acid and a very few crystals of potassium chlorate. Warm gently for two minutes and then apply tests for ferrous and for ferric ions. What are the results? Write an equation for the reaction that takes place.

c. Prepare a dilute solution of ferrous sulfate by dissolving 0.5 g. of the salt in 10 ml. of water. Divide the solution into two equal portions. Now acidify with dilute sulfuric acid 10 ml. of a solution of potassium permanganate and add it, drop by drop, to one portion of the ferrous sulfate solution. Continue the addition as long as the permanganate solution is decolorized. Test separate portions of the solution for ferric and ferrous ions. What result is observed? Write an equation to express the oxidation of the ferrous salt.

d. Repeat *c* with the remaining portion of the ferrous sulfate solution prepared in *c*, using acidulated potassium dichromate solution in place of potassium permanganate. What is the equation for the reaction?

233. Reduction of Ferric Salts. *a*. Acidify 3 ml. of a ferric chloride solution with an equal volume of dilute hydrochloric acid. Add several grams of iron filings and allow the reaction to go on for five minutes. Filter off the undissolved iron, and test the filtrate for ferrous and for ferric ions. What result is observed? Repeat, using powdered zinc in place of iron. What result is observed? Write equations for the reactions. What are the reducing agents in these instances? Which chloride of iron should you expect to obtain when iron is dissolved in hydrochloric acid? Confirm your prediction.

b. Fit up a small Erlenmeyer flask with a one-hole rubber stopper and delivery tube. Generate hydrogen in the flask by the action of dilute hydrochloric acid on granulated zinc, and bubble the evolved gas through 3 ml. of ferric chloride solution. After three minutes test the solution for ferrous iron. What is the result? Account for the difference observed in the action of hydrogen in *a.*

c. Saturate with hydrogen sulfide 3 ml. of ferric chloride solution (*HOOD!*). What is the precipitate? Test the solution for ferrous and for ferric ions. What is the result? Write an equation for the oxidation-reduction reaction.

d. Determine whether or not stannous chloride will reduce a solution of a ferric salt.

e. Certain ferric salts are reduced to the ferrous condition on exposure to light. Consult your text on the subject of "blue prints."

234. Complex Cyanides of Iron. *a.* Add a few drops of a solution of potassium cyanide (*CARE!*) to 3 ml. of a solution of ferrous chloride. Note the color of the precipitate that is formed. What is the compound? Now continue the addition of potassium cyanide solution until the precipitate just dissolves. What is present in the solution now? Confirm your prediction by testing the solution with a few drops of a solution of ferric chloride acidified with acetic acid. What is the result? Write equations for the reactions that have taken place in this experiment.

b. Add a solution of potassium cyanide to 3 ml. of ferric chloride solution until the precipitate first formed just dissolves. What is the precipitate? What does the solution now contain? What should be the action of a ferric salt on this solution? What reaction should take place on the addition of a ferrous salt? Acidify with acetic acid solutions of ferric and of ferrous salts and test the correctness of your answers. Write equations for all reactions taking place.

235. Preparation of Ferrous Ammonium Sulfate. *a.* Dissolve 5 g. of iron filings in 15 ml. of dilute sulfuric acid. Filter the solution. What is the residue? What weight of ferrous sulfate should be in the filtrate? Crystals of ferrous ammonium sulfate contain one molecule of ammonium sulfate for each molecule of ferrous sulfate present. From this fact calculate the quantity of ammonium sulfate necessary to form ferrous ammonium sulfate with the ferrous sulfate prepared from 5 g. of iron.

Place your ferrous sulfate solution in a 50-milliliter beaker and dilute it with 10 ml. of water. Heat the solution to 80° and dissolve in it the calculated weight of ammonium sulfate crystals. Cover the beaker with a watch glass and set aside in your desk until the next laboratory period. At this time filter off the crystals that have formed and dry them between filter papers. What weight of the compound have you obtained? What is the theoretical yield of $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6\,H_2O$? What per cent of the theoretical yield was obtained in this experiment?

b. Obtain experimental evidence to show that crystals of ferrous ammonium sulfate contain water of hydration. Show that a solution of the compound yields ferrous ions and sulfate ions. How could you prove the ammonium ion to be present in the solution? Verify your answer.

236. Preparation of Ferric Ammonium Alum. Prepare 30 ml. of dilute sulfuric acid by adding 5 ml. of the concentrated acid to 25 ml. of water. Place the dilute acid in a 50-milliliter beaker and add 20 g. of ferrous sulfate crystals, $FeSO_4 \cdot 7\,H_2O$. Stir and heat to boiling. Now add slowly, and with constant stirring, 7 ml. of concentrated nitric acid. After the addition of the acid boil the solution for five minutes (***HOOD!***). Consult Experiment 232, *a*, for the reaction that takes place. What weight of ferric sulfate is contained in the solution you have just prepared?

Look up the formula of ferric ammonium sulfate and calculate the weight of ammonium sulfate necessary for the formation of the alum from the quantity of ferric sulfate prepared in this experiment. Obtain the calculated amount of solid ammonium sulfate, dissolve it in 20 ml. of boiling water, and add it to your solution of ferric sulfate. Filter into a beaker, cover with a watch glass, and set it aside in your desk until the next laboratory period. Filter off the crystals and dry them between filter papers.

What is the general formula for an alum? Write the formula for the one you have prepared. What weight of the alum have you obtained? What is the theoretical yield of alum obtainable from the quantity of ferric sulfate you prepared? Calculate the percentage yield.

237. Reactions of Cobalt Compounds. *a.* Heat a few crystals of cobalt nitrate in a small porcelain crucible. What change occurs? What remains in the crucible? Touch a hot borax bead to a minute bit of the residue and heat strongly in the oxidizing flame of the blowpipe. What is the result? The color developed affords a delicate test for cobalt.

b. Acidify 5 ml. of cobalt nitrate solution with acetic acid. Prepare an equal volume of a saturated solution of potassium nitrite and add it to the solution of cobalt nitrate. The formation of a yellow precipitate on standing is a test often used for the qualitative detection of cobalt. Write an equation for the reaction that takes place.

c. Add a solution of potassium cyanide, drop by drop, to 2 ml. of a solution of cobalt salt until the precipitate first formed dissolves in an excess of the reagent. Write equations for the reactions. Now add 2 ml. of sodium hydroxide solution, and boil. To the boiling solution add bromine water, drop by drop, until the color of the bromine persists. What is the reaction that occurs?

d. Add a few drops of an alcoholic solution of dimethylglyoxime to 2 ml. of a solution of a cobalt salt. Make the solution slightly ammoniacal and then boil it. If the cobalt salt is pure (free from nickel), a precipitate will not be formed.

238. Reactions of Nickel Compounds. *a.* Repeat Experiment 237, *a,* using a nickel salt. Note the color of the bead. Make another bead, and dip it into a mixture containing two drops of cobalt nitrate solution in 5 ml. of nickel nitrate solution. What color develops on heating the bead? A small amount of cobalt is sufficient to mask the brown coloration produced by nickel.

b. Repeat Experiment 237, *c,* using nickel nitrate solution. Note the difference in results. Write equations for the reactions that take place. The formation of the black precipitate furnishes a means for the detection of nickel in the presence of cobalt. What is the black precipitate?

c. Repeat Experiment 237, *d*, using a solution of a nickel salt. What is the result? What is the characteristically colored precipitate? The formation of this substance constitutes a very delicate qualitative test for nickel.

EXERCISES

1. What are the usual ores of iron?
2. How is iron obtained from its ores?
3. Distinguish between iron and steel.
4. Should you expect a red color when you added a solution of ammonium thiocyanate to one of potassium ferricyanide? Explain.
5. What are the usual ores of nickel?
6. What are the usual ores of cobalt?
7. Do cobalt and nickel form complex salts?

CHAPTER XLIV

SPECIAL PREPARATIONS

239. Preparation of Ammonium Thiocyanate (NH_4CNS). Mix together 60 g. of 95-per-cent alcohol, 80 g. of ammonium hydroxide (specific gravity 0.912), and 40 g. of carbon disulfide. Allow the mixture to stand for four days in a stoppered flask (*CARE! HOOD!*). Concentrate the liquid to about half its volume by heating it in an evaporating dish on a water bath. Set the dish aside to allow crystallization of the salt. Collect the crystals on a funnel. Wash repeatedly with alcohol. Dry and weigh them. What per cent of the theoretical yield did you obtain? Give equations for the formation of the salt.

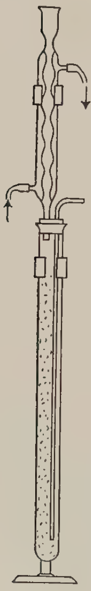

FIG. 60

240. Preparation of Stannic Chloride ($SnCl_4$). Seal off one end of a glass tube 3 or 4 cm. in diameter and about 40 cm. long. Fit the open end of the tube with a two-hole rubber stopper carrying a reflux condenser and a long glass tube reaching nearly to the bottom of the large tube (Fig. 60). Fill the large tube nearly two thirds full of granulated tin, connect the delivery tube with a chlorine tank or generator, and pass in *dry* chlorine. When liquid stannic chloride forms, adjust the delivery tube so that it just reaches below the level of the liquid. Continue to pass in chlorine until the tin is nearly all used up. Disconnect the apparatus and purify the stannic chloride by distillation. Keep it in a sealed tube.

241. Preparation of Sodium Cobaltinitrite ($Na_3Co(NO_2)_6$). Dissolve 5 g. of cobaltous nitrate and 15 g. of sodium nitrite in 15 ml. of hot water. Cool the resulting solution to 40° and, with frequent shaking of the liquid, add to it, drop by drop, 5 ml. of 50-per-cent acetic acid. Draw air through the mixture for half an hour and set it aside until the next laboratory period. Filter off any precipitate and treat it (the precipitate) with 5 ml. of water heated to 80°. Filter and combine this filtrate with the previous one. Stir into the combined filtrates 35 ml. of 95-per-cent alcohol. After allowing the mixture to stand for an hour or two, collect the precipitate on a filter and wash it twice with alcohol. Allow the salt to dry and then weigh it. What is the yield?

242. Preparation of Chloropentammine Cobaltic Chloride ($Co(NH_3)_5Cl_3$). Add dilute hydrochloric acid, a little at a time, to 10 g. of cobalt carbonate until no further action takes place. Filter the solution, if necessary, and add 125 ml. of 10-per-cent ammonium hydroxide, followed by a solution of 25 g. of ammonium carbonate in 125 ml. of water. Draw air through this mixture for three hours. Add to the resulting solution 75 g. of ammonium chloride and evaporate the mixture on a water bath to a pasty mass. Stir in dilute hydrochloric acid, a little at a time, until no more carbon dioxide is evolved. Make the solution just alkaline with ammonium hydroxide and then add 10 ml. excess of the same reagent. Dilute this solution to a volume of about 250 ml. and heat it on a water bath for one hour. Add to the solution 150 ml. of concentrated hydrochloric acid and continue the heating for about three quarters of an hour. Allow the mixture to become cold. Crystals of chloropentammine cobaltic chloride will gradually separate out during the heating and cooling. Filter out these crystals on a suction filter and wash them with very dilute hydrochloric acid. Finally wash the precipitate several times with alcohol. Dry the crystals on an air bath and present them for inspection.

243. Preparation of Chromous Acetate ($Cr(C_2H_3O_2)_2$). Powder 25 g. of potassium dichromate and dissolve it in 100 ml. of water in a 500-milliliter flask. Add to the solution 75 ml. of concentrated hydrochloric acid. Place the flask under the hood and add 15 ml. of alcohol. Shake the mixture, if necessary, to start the action. Allow the mixture to stand until the action has ceased and then reduce its volume one half by boiling the solution. What color is the solution? What ion produces this color? Some solid potassium chloride may separate out when the solution cools.

Pour the solution upon 70 g. of granulated zinc in a second 500-milliliter flask. Add to the mixture 75 ml. of concentrated hydrochloric acid (*HOOD!*). When the first violent action is over, place in the neck of the flask a rubber stopper carrying two tubes, one ending just below the stopper, the other extending nearly to the bottom of the flask. Connect the latter with a delivery tube reaching below the surface of a solution of 100 g. of sodium acetate in 500 ml. of water (Fig. 61). When the chromium solution becomes a clear sky-blue, place your finger over the outer end of the shorter tube. The chromium solution will thus be forced over into the solution of sodium acetate. When most of the material has passed over, remove your finger. Allow the precipitate to settle. Siphon off the supernatant liquid, wash the precipitate several times with water charged with carbon dioxide, and siphon off the wash liquid. Preserve the chromous acetate as a wet paste in a tightly stoppered bottle.

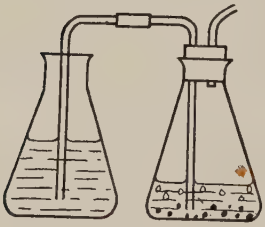

FIG. 61

244. Preparation of Sodium Thiocarbonate ($Na_2CS_3 \cdot H_2O$). Place 25 ml. of 1-*N* sodium hydroxide in a 250-milliliter flask and saturate the solution with hydrogen sulfide. Add to this solution 25 ml. of 1-*N* sodium hydroxide. Pour 10 ml. of carbon disulfide into the flask and shake it vigorously at five-minute intervals for one hour. Separate any undissolved carbon disulfide by the use of a separatory funnel. Treat the solution with an equal volume of ether. Shake the flask several times and allow the mixture to stand until the crystals of thiocarbonate settle out. Decant the mother liquor and wash the crystals with ether by decantation.

Dissolve 0.3 g. of these crystals in water and make the solution acid by adding to it, a drop at a time, dilute hydrochloric acid. Note the heavy red oil which separates out. It is perthiocarbonic acid.

245. The Preparation of Chrome Red. Fuse 15 g. of potassium nitrate in a 30-milliliter porcelain crucible. Now heat the crucible strongly until a dull red heat is reached, and then add lead chromate in 0.5-gram portions until a total weight of 5 g. has been added. The reaction causes foaming to take place so that it is necessary to wait after each addition of lead chromate until the foam has subsided. Care must be taken not to heat the mixture too strongly, because this results in a dark-brown product. After all the lead chromate has been added, allow the contents of the crucible to cool to about 100°, and then slowly add 25 ml. of water. Now transfer the contents of the crucible to a mortar and thoroughly crush all the undissolved material. After allowing the solid material to settle, decant the clear yellow solution. The chrome red should then be washed into a filter, and there leached out with cold water until the washings run through colorless. Then remove the filter paper with its contents from the funnel, and allow the chrome red to dry on the paper at room temperature. The dry product is a bright-red insoluble compound, $PbCrO_4 \cdot PbO$.

246. Additional Preparations. Consult the literature, and decide upon a method for preparing each of the following substances: antimony trichloride; trisodium phosphate; microcosmic salt; selenic acid; strontium chloride and strontium hydroxide from strontium sulfate; barium nitrate from barium sulfate; aluminum sulfate from aluminum oxide; mosaic gold; manganese dioxide (water-free); sodium thiosulfate; mercuric thiocyanate. Before carrying out any one of these preparations, submit your method to your instructor for his approval.

APPENDIX

TABLE I. SOLUBILITIES OF SOME INORGANIC SUBSTANCES IN WATER AT 18°

SUBSTANCE	$\frac{\text{GRAM}}{100}$	C_M	SUBSTANCE	$\frac{\text{GRAM}}{100}$	C_M
Barium bromide	103.6	2.9	Silver bromide	$0.0_{4}1$	$0.0_{6}6$
Barium carbonate	$0.0_{2}22$	$0.0_{3}11$	Silver carbonate	$0.0_{2}3$	$0.0_{3}1$
Barium chlorate	35.48	1.1	Silver chlorate	12.25	0.6
Barium chloride	35.2	1.7	Silver chloride	$0.0_{3}15$	$0.0_{5}9$
Barium chromate	$0.0_{3}38$	$0.0_{4}15$	Silver chromate	$0.0_{2}24$	$0.0_{3}15$
Barium fluoride	0.161	$0.0_{2}92$	Silver fluoride	195.4	6.75
Barium hydroxide	3.7	0.216	Silver iodide	$0.0_{6}35$	$0.0_{7}1$
Barium iodide	201.36	3.8	Silver nitrate	211.6	8.3
Barium nitrate	8.8	0.33	Silver oxide	$0.0_{2}2$	$0.0_{4}8$
Barium sulfate	$0.0_{3}23$	$0.0_{4}1$	Silver sulfate	0.58	0.021
Calcium bromide	142.	5.2	Sodium bromide	89.1	6.9
Calcium carbonate	$0.0_{2}13$	$0.0_{3}13$	Sodium carbonate	19.3	1.8
Calcium chlorate	179.3	5.3	Sodium chlorate	98.6	6.5
Calcium chloride	71.	5.25	Sodium chloride	35.9	5.42
Calcium chromate	2.3	0.147	Sodium chromate	66.9	3.54
Calcium fluoride	$0.0_{2}16$	$0.0_{3}2$	Sodium fluoride	4.49	1.07
Calcium hydroxide	0.17	0.022	Sodium hydroxide	107.5	20.00
Calcium iodide	202.8	4.86	Sodium iodide	176.7	8.00
Calcium nitrate	125.8	5.37	Sodium nitrate	86.1	7.5
Calcium sulfate	0.20	0.011	Sodium sulfate	16.9	1.15
Magnesium bromide	103.2	4.6	Strontium bromide	96.5	3.4
Magnesium carbonate	0.1	0.011	Strontium carbonate	0.0011	$0.0_{4}75$
Magnesium chlorate	128.8	4.78	Strontium chlorate	172.5	4.5
Magnesium chloride	56.00	5.1	Strontium chloride	52.7	3.1
Magnesium chromate	72.8	4.3	Strontium chromate	0.12	$0.0_{2}59$
Magnesium fluoride	$0.0_{2}87$	$0.0_{2}14$	Strontium fluoride	0.012	$0.0_{3}93$
Magnesium hydroxide	$0.0_{2}1$	$0.0_{3}2$	Strontium hydroxide	0.77	0.0633
Magnesium iodide	148.1	4.1	Strontium iodide	173.9	4.00
Magnesium nitrate	73.4	3.7	Strontium nitrate	67.6	2.7
Magnesium sulfate	35.2	2.8	Strontium sulfate	0.011	$0.0_{3}62$
Potassium bromide	64.1	4.7	Zinc bromide	478.2	9.8
Potassium carbonate	111.00	6.4	Zinc carbonate	0.005	$0.0_{3}4$
Potassium chlorate	6.7	0.53	Zinc chlorate	198.7	5.7
Potassium chloride	33.9	4.0	Zinc chloride	361(?)	(?)
Potassium chromate	62.5	2.7	Zinc fluoride	1.6	0.15
Potassium fluoride	92.3	12.36	Zinc hydroxide	$0.0_{3}42$	$0.0_{4}4$
Potassium hydroxide	110.00	14.6	Zinc iodide	431.9	6.1
Potassium iodide	142.6	6.2	Zinc nitrate	115.00	4.6
Potassium nitrate	29.1	2.3	Zinc sulfate	55.6	3.4
Potassium sulfate	10.7	0.6	Zinc sulfide	$0.0_{10}33$	$0.0_{11}3$

TABLE II. INTERNATIONAL ATOMIC WEIGHTS (1927)

Name of Element	Symbol	Atomic Number	Atomic Weight	Name of Element	Symbol	Atomic Number	Atomic Weight
Aluminum	Al	13	26.97	Mercury	Hg	80	200.61
Antimony	Sb	51	121.77	Molybdenum	Mo	42	96.0
Argon	A	18	39.91	Neodymium	Nd	60	144.27
Arsenic	As	33	74.96	Neon	Ne	10	20.2
Barium	Ba	56	137.37	Nickel	Ni	28	58.69
Beryllium	Be	4	9.02	Nitrogen	N	7	14.008
Bismuth	Bi	83	209.00	Osmium	Os	76	190.8
Boron	B	5	10.82	Oxygen	O	8	16.000
Bromine	Br	35	79.916	Palladium	Pd	46	106.7
Cadmium	Cd	48	112.41	Phosphorus	P	15	31.027
Cæsium	Cs	55	132.81	Platinum	Pt	78	195.23
Calcium	Ca	20	40.07	Potassium	K	19	39.096
Carbon	C	6	12.000	Praseodymium	Pr	59	140.92
Cerium	Ce	58	140.25	Radium	Ra	88	225.95
Chlorine	Cl	17	35.457	Radon	Rn	86	222.
Chromium	Cr	24	52.01	Rhodium	Rh	45	102.91
Cobalt	Co	27	58.94	Rubidium	Rb	37	85.44
Columbium	Cb	41	93.1	Ruthenium	Ru	44	101.7
Copper	Cu	29	63.57	Samarium	Sm	62	150.43
Dysprosium	Dy	66	162.52	Scandium	Sc	21	45.10
Erbium	Er	68	167.7	Selenium	Se	34	79.2
Europium	Eu	63	152.0	Silicon	Si	14	28.06
Fluorine	F	9	19.00	Silver	Ag	47	107.880
Gadolinium	Gd	64	157.26	Sodium	Na	11	22.997
Gallium	Ga	31	69.72	Strontium	Sr	38	87.63
Germanium	Ge	32	72.60	Sulfur	S	16	32.064
Gold	Au	79	197.2	Tantalum	Ta	73	181.5
Hafnium	Hf	72	178.6	Tellurium	Te	52	127.5
Helium	He	2	4.00	Terbium	Tb	65	159.2
Holmium	Ho	67	163.5	Thallium	Tl	81	204.39
Hydrogen	H	1	1.008	Thorium	Th	90	232.15
Indium	In	49	114.8	Thulium	Tu	69	169.4
Iodine	I	53	126.932	Tin	Sn	50	118.70
Iridium	Ir	77	193.1	Titanium	Ti	22	47.90
Iron	Fe	26	55.84	Tungsten	W	74	184.0
Krypton	Kr	36	82.9	Uranium	U	92	238.17
Lanthanum	La	57	138.90	Vanadium	V	23	50.96
Lead	Pb	82	207.22	Xenon	Xe	54	130.2
Lithium	Li	3	6.940	Ytterbium	Yb	70	173.6
Lutecium	Lu	71	175.0	Yttrium	Y	39	89.0
Magnesium	Mg	12	24.32	Zinc	Zn	30	65.38
Manganese	Mn	25	54.93	Zirconium	Zr	40	91.22

TABLE III. PRESSURE IN MILLIMETERS OF MERCURY OF SATURATED AQUEOUS VAPOR

Temperature	Pressure in Millimeters	Temperature	Pressure in Millimeters	Temperature	Pressure in Millimeters
0	4.58	16	13.64	32	35.70
1	4.92	17	14.54	33	37.78
2	5.29	18	15.49	34	39.95
3	5.68	19	16.49	35	42.19
4	6.10	20	17.55	40	55.40
5	6.54	21	18.66	45	71.97
6	7.01	22	19.84	50	92.60
7	7.51	23	21.09	60	149.60
8	8.05	24	22.40	70	233.90
9	8.61	25	23.78	80	355.47
10	9.21	26	25.24	90	526.00
11	9.85	27	26.77	100	760.00
12	10.52	28	28.38	150	3,568.7
13	11.24	29	30.08	200	11,647.
14	11.99	30	31.86	250	29,771.
15	12.79	31	33.74	300	64,290.

TABLE IV. SOLID REAGENTS

Acid, boric
Acid, oxalic
Acid, picric
Acid, tartaric
Agar-agar
Albumin
Alizarin
Aluminum (metal)
Aluminum chloride
Aluminum nitrate
Aluminum oxide
Aluminum sulfate
Ammonium acetate
Ammonium carbonate
Ammonium chloride
Ammonium nitrate
Ammonium oxalate
Ammonium persulfate
Ammonium phosphate
Ammonium sulfate
Ammonium thiocyanate
Antimony (metal)
Antimony trichloride
Antimony trioxide
Arsenic (metal)
Arsenic trioxide
Arsenic trisulfide
Asbestos
Barium carbonate
Barium chloride
Barium hydroxide
Barium nitrate
Barium oxide
Barium peroxide
Barium sulfate
Beta-naphthol
Bismuth (metal)
Bismuth chloride
Bismuth nitrate
Bismuth oxide
Bleaching powder (chloride of lime)
Cadmium chloride
Cadmium nitrate
Cadmium sulfate
Calcium (metal; *storeroom*)
Calcium carbide
Calcium carbonate (marble; limestone)

Calcium carbonate (powder)
Calcium chloride (anhydrous)
Calcium chloride (crystals)
Calcium fluoride
Calcium hydroxide (slaked lime)
Calcium nitrate
Calcium oxide (quicklime)
Calcium phosphate
Calcium phosphide
Calcium sulfate
Calico
Cement (Portland)
Charcoal (animal)
Charcoal (wood)
Chrome alum
Chromic acetate
Chromic nitrate
Chromium oxide
Coal
Cobalt carbonate
Cobalt chloride
Cobalt nitrate
Cobalt oxide
Cobalt sulfate
Copper (metal)
Copper (wire)
Copper (wool)
Cotton
Cupric bromide
Cupric chloride
Cupric nitrate
Cupric oxide
Cupric oxide (wire)
Cupric sulfate
Cuprous oxide
Ferric chloride
Ferric oxide
Ferric sulfate
Ferrous ammonium sulfate
Ferrous sulfate
Ferrous sulfide
Gelatin
Gold leaf
Graphite
Iodine (crystals)
Iron filings
Iron nails (eightpenny)
Iron nails (shingle)
Iron pyrites
Iron wire (30–40 gauge)
Lead (metal)
Lead carbonate
Lead chromate
Lead dioxide
Lead monoxide (litharge)
Lead nitrate
Lead sulfide
Litmus papers
Magnesium (metal; ribbon)
Magnesium carbonate (magnesite)
Magnesium chloride
Magnesium nitrate
Magnesium oxide
Magnesium sulfate
Manganese dioxide
Manganous chloride
Manganous sulfate
Mercury (metal)
Mercuric chloride
Mercuric iodide
Mercuric nitrate
Mercuric oxide
Mercuric sulfide
Mercurous chloride
Mercurous nitrate
Nickel chloride
Nickel nitrate
Nickel oxide
Nickel sulfate
Paraffin
Pectin
Phosphorus (red)
Phosphorus (white; *storeroom*)
Phosphorus pentoxide
Potassium (*storeroom*)
Potassium aluminum sulfate
Potassium antimonyl tartrate
Potassium bromate
Potassium bromide
Potassium carbonate
Potassium chlorate
Potassium chloride
Potassium chromate
Potassium chromium sulfate

Potassium cyanide
Potassium dichromate
Potassium ferricyanide
Potassium ferrocyanide
Potassium hydrogen sulfate
Potassium hydrogen tartrate
Potassium hydroxide
Potassium iodate
Potassium iodide
Potassium iodide starch papers
Potassium nitrate
Potassium nitrite
Potassium perchlorate
Potassium permanganate
Potassium persulfate
Potassium sulfate
Potassium thiocyanate
Pyrogallic acid
Red lead
Rosin
Sawdust
Silicon
Silicon dioxide
Silver nitrate
Soap
Soda lime
✓ Sodium (metal; *storeroom*)
Sodium acetate
Sodium ammonium hydrogen phosphate (microcosmic salt)
Sodium bromide
Sodium carbonate
Sodium chlorate
Sodium chloride (commercial)
Sodium chloride (rock salt)
Sodium dichromate
Sodium fluoride
Sodium hydrogen carbonate
Sodium hydrogen sulfate
Sodium hydrogen sulfite
Sodium hydroxide
Sodium iodide
Sodium nitrate
Sodium nitrite
Sodium nitroprusside
Sodium oxalate
Sodium peroxide (*storeroom*)
Sodium phosphate (primary)
Sodium phosphate (secondary)
Sodium phosphate (tertiary)
Sodium potassium tartrate
Sodium pyrophosphate
Sodium silicate
Sodium stannate
Sodium sulfate
Sodium sulfite
Sodium tetraborate (borax)
Sodium thiosulfate
Soil
Solder (rosin centered)
Stannic chloride
Stannous chloride
Starch
✓ Sterling silver
Strontium chloride
Strontium nitrate
Strontium sulfate
Sugar
Sulfur (flowers)
Sulfur (roll)
Talc
✓ Tin (metal)
Vaseline
✓ Zinc (metal; granular)
Zinc (dust)
Zinc chloride
Zinc oxide
Zinc sulfate
Zinc sulfide

TABLE V. SOLUTIONS AND LIQUIDS

Name	Concentration
Acetic acid (dilute)	6-*N*
Acetic acid (glacial)	99 per cent
Boric acid	Saturated
Oxalic acid	2-*N*
Perchloric acid	2 per cent
Phosphoric acid	85 per cent
Picric acid	Saturated
Tartaric acid	1-*N*
Acetone	—
Alcohol (*storeroom*)	—
Aluminum chloride	1-*N*
Aluminum nitrate	1-*N*
Aluminum sulfate	1-*N*
Ammonium acetate	2-*N*
Ammonium carbonate	1-*N*
Ammonium chloride	4-*N*
Ammonium molybdate (HNO_3)	(See special reagents)
Ammonium nitrate	4-*N*
Ammonium oxalate	2-*N*
Ammonium phosphate	1-*N*
Ammonium sulfate	1-*N*
Ammonium sulfide	3-*N*
Ammonium sulfide, yellow	3-*N*
Ammonium thiocyanate	1-*N*
Antimony trichloride	1-*N*
Auric chloride (*storeroom*)	—
Barium chloride	1-*N*
Barium hydroxide	4-*N*
Barium nitrate	1-*N*
Bismuth nitrate	1-*N*
Bromine	—
Bromine water	Saturated
Cadmium chloride	1-*N*
Cadmium nitrate	1-*N*
Cadmium sulfate	1-*N*
Calcium chloride	1-*N*
Calcium hydroxide (limewater)	Saturated
Calcium nitrate	1-*N*
Calcium sulfate	Saturated
Carbon disulfide	—
Carbon tetrachloride	—
Chlorine water	Saturated
Chromium nitrate	1-*N*
Chromium sulfate	1-*N*
Cobalt chloride	1-*N*
Cobalt nitrate	1-*N*
Cupric nitrate	1-*N*
Cupric sulfate	1-*N*
Ether	—
Ferric chloride	1-*N*
Ferric sulfate	1-*N*
Ferrous sulfate	2-*N*
Glucose	—
Hydrogen peroxide (*storeroom*)	—
Indigo solution	—
Iodine water	Saturated
Kerosene	—
Lead acetate	1-*N*
Lead nitrate	1-*N*
Litmus	0.1-*N*
Magnesia mixture	(See special reagents)
Magnesium chloride	2-*N*
Magnesium sulfate	0.5-*N*
Manganous chloride	1-*N*
Manganous sulfate	1-*N*
Mercuric chloride	1-*N*
Mercuric nitrate	1-*N*
Mercurous nitrate	1-*N*
Methyl alcohol	—
Methyl orange	10 g. per liter
Methyl violet	8 g. per liter
Nickel nitrate	1-*N*
Nickel sulfate	1-*N*
Phenolphthalein	½ g. per liter
Phloroglucinol	1 per cent
Phosphorus trichloride	—
Potassium aluminum sulfate	1-*N*
Potassium bromate	0.5-*N*
Potassium bromide	1-*N*
Potassium chlorate	0.5-*N*
Potassium chloride	1-*N*
Potassium chromate	3-*N*
Potassium chromium sulfate	1-*N*
Potassium cyanide	0.5-*N*
Potassium dichromate	1-*N*
Potassium ferricyanide	0.5-*N*
Potassium ferrocyanide	0.5-*N*
Potassium hydroxide	2-*N*
Potassium iodide	0.1-*N*
Potassium nitrate	1-*N*
Potassium nitrite	1-*N*
Potassium permanganate	0.5-*N*
Potassium permanganate (acid; *storeroom*)	(See special reagents)
Potassium pyroantimonate	(See special reagents)
Silver nitrate	0.2-*N*
Silver sulfate	Saturated
Sodium acetate	1-*N*
Sodium arsenate	1-*N*
Sodium arsenite	1-*N*
Sodium carbonate	2-*N*
Sodium chloride	1-*N*
Sodium cobaltinitrite	1-*N*
Sodium hydrogen carbonate	Saturated
Sodium hydrogen sulfate	Saturated
Sodium hydrogen sulfide	3-*N*
Sodium hydrogen tartrate	Saturated
Sodium phosphate, primary	0.5-*N*
Sodium phosphate, secondary	0.5-*N*
Sodium phosphate, tertiary	0.5-*N*
Sodium potassium tartrate	1-*N*
Sodium sulfate	1-*N*
Sodium tetraborate	1-*N*
Sodium thiosulfate	1-*N*
Stannous chloride	0.5-*N*
Starch emulsion	—
Strontium chloride	1-*N*
Strontium sulfate	Saturated
Zinc acetate	0.5-*N*
Zinc chloride	1-*N*
Zinc nitrate	1-*N*

TABLE VI. DESK REAGENTS

Ammonium hydroxide	6-N
Hydrochloric acid (dilute)	6-N
Hydrochloric acid (concentrated)	12-N
Nitric acid (dilute)	6-N
Nitric acid (concentrated)	16-N
Sodium hydroxide	6-N
Sulfuric acid (dilute)	6-N
Sulfuric acid (concentrated)	95 per cent

TABLE VII. SPECIAL REAGENTS

Dimethylglyoxime. Dissolve 12 g. of dimethylglyoxime in 1000 ml. of 95-per-cent alcohol.

Ferrous sulfate. Make a saturated solution of ferrous sulfate in 0.6-N sulfuric acid. Keep iron nails in the solution.

Magnesia mixture. Dissolve 100 g. of ammonium chloride and 100 g. of magnesium chloride in water and add to the solution 5 ml. of concentrated ammonium hydroxide. Dilute the solution to 1000 ml.

Nitric acid solution of ammonium molybdate. 150 g. of ammonium molybdate is dissolved in enough water to make a liter of solution; this is added to a liter of 6-N nitric acid, and 300 g. of ammonium nitrate is added to the mixture.

Potassium pyroantimonate. 20 g. of the salt is added to 1000 ml. of boiling water, and the mixture is kept at boiling until the substance dissolves. Quickly cool the solution, add to it 80 ml. of a 10-per-cent solution of potassium hydroxide, and filter.

Sodium cobaltinitrite. Use the material prepared in Experiment 241.

Stannous chloride solution. Dissolve 115 g. of stannous chloride in 170 ml. of concentrated hydrochloric acid. Dilute the solution with water to 1 liter and add granulated tin.

Acid permanganate solution. Dissolve 0.5 g. of potassium permanganate in 2000 ml. of distilled water and add to the solution (*CARE!*) 130 ml. of concentrated sulfuric acid.

Starch paste. Grind 1 g. of starch in a mortar. Make the starch into a thin paste with cold water. Pour this into 1 liter of boiling water, continue the boiling for one minute, and allow the mixture to cool.

Sodium stannite. Add sodium hydroxide to a freshly prepared solution of stannous chloride until the precipitate which first forms is dissolved.

TABLE VIII. TABLE OF LOGARITHMS

First Two Figures	Third Figure										Fourth-Figure Differences								
	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9
10	0000	0043	0086	0128	0170	0212	0253	0294	0334	0374	4	8	12	17	21	25	29	33	37
11	0414	0453	0492	0531	0569	0607	0645	0682	0719	0755	4	8	11	15	19	23	26	30	34
12	0792	0828	0864	0899	0934	0969	1004	1038	1072	1106	3	7	10	14	17	21	24	28	31
13	1139	1173	1206	1239	1271	1303	1335	1367	1399	1430	3	6	10	13	16	19	23	26	29
14	1461	1492	1523	1553	1584	1614	1644	1673	1703	1732	3	6	9	12	15	18	21	24	27
15	1761	1790	1818	1847	1875	1903	1931	1959	1987	2014	3	6	8	11	14	17	20	22	25
16	2041	2068	2095	2122	2148	2175	2201	2227	2253	2279	3	5	8	11	13	16	18	21	24
17	2304	2330	2355	2380	2405	2430	2455	2480	2504	2529	2	5	7	10	12	15	17	20	22
18	2553	2577	2601	2625	2648	2672	2695	2718	2742	2765	2	5	7	9	12	14	16	19	21
19	2788	2810	2833	2856	2878	2900	2923	2945	2967	2989	2	4	7	9	11	13	16	18	20
20	3010	3032	3054	3075	3096	3118	3139	3160	3181	3201	2	4	6	8	11	13	15	17	19
21	3222	3243	3263	3284	3304	3324	3345	3365	3385	3404	2	4	6	8	10	12	14	16	18
22	3424	3444	3464	3483	3502	3522	3541	3560	3579	3598	2	4	6	8	10	12	14	15	17
23	3617	3636	3655	3674	3692	3711	3729	3747	3766	3784	2	4	6	7	9	11	13	15	17
24	3802	3820	3838	3856	3874	3892	3909	3927	3945	3962	2	4	5	7	9	11	12	14	16
25	3979	3997	4014	4031	4048	4065	4082	4099	4116	4133	2	3	5	7	9	10	12	14	15
26	4150	4166	4183	4200	4216	4232	4249	4265	4281	4298	2	3	5	7	8	10	11	13	15
27	4314	4330	4346	4362	4378	4393	4409	4425	4440	4456	2	3	5	6	8	9	11	13	14
28	4472	4487	4502	4518	4533	4548	4564	4579	4594	4609	2	3	5	6	8	9	11	12	14
29	4624	4639	4654	4669	4683	4698	4713	4728	4742	4757	1	3	4	6	7	9	10	12	13
30	4771	4786	4800	4814	4829	4843	4857	4871	4886	4900	1	3	4	6	7	9	10	11	13
31	4914	4928	4942	4955	4969	4983	4997	5011	5024	5038	1	3	4	6	7	8	10	11	12
32	5051	5065	5079	5092	5105	5119	5132	5145	5159	5172	1	3	4	5	7	8	9	11	12
33	5185	5198	5211	5224	5237	5250	5263	5276	5289	5302	1	3	4	5	6	8	9	10	12
34	5315	5328	5340	5353	5366	5378	5391	5403	5416	5428	1	3	4	5	6	8	9	10	11
35	5441	5453	5465	5478	5490	5502	5514	5527	5539	5551	1	2	4	5	6	7	9	10	11
36	5563	5575	5587	5599	5611	5623	5635	5647	5658	5670	1	2	4	5	6	7	8	10	11
37	5682	5694	5705	5717	5729	5740	5752	5763	5775	5786	1	2	3	5	6	7	8	9	10
38	5798	5809	5821	5832	5843	5855	5866	5877	5888	5899	1	2	3	5	6	7	8	9	10
39	5911	5922	5933	5944	5955	5966	5977	5988	5999	6010	1	2	3	4	5	7	8	9	10
40	6021	6031	6042	6053	6064	6075	6085	6096	6107	6117	1	2	3	4	5	6	8	9	10
41	6128	6138	6149	6160	6170	6180	6191	6201	6212	6222	1	2	3	4	5	6	7	8	9
42	6232	6243	6253	6263	6274	6284	6294	6304	6314	6325	1	2	3	4	5	6	7	8	9
43	6335	6345	6355	6365	6375	6385	6395	6405	6415	6425	1	2	3	4	5	6	7	8	9
44	6435	6444	6454	6464	6474	6484	6493	6503	6513	6522	1	2	3	4	5	6	7	8	9
45	6532	6542	6551	6561	6571	6580	6590	6599	6609	6618	1	2	3	4	5	6	7	8	9
46	6628	6637	6646	6656	6665	6675	6684	6693	6702	6712	1	2	3	4	5	6	7	7	8
47	6721	6730	6739	6749	6758	6767	6776	6785	6794	6803	1	2	3	4	5	5	6	7	8
48	6812	6821	6830	6839	6848	6857	6866	6875	6884	6893	1	2	3	4	4	5	6	7	8
49	6902	6911	6920	6928	6937	6946	6955	6964	6972	6981	1	2	3	4	4	5	6	7	8
50	6990	6998	7007	7016	7024	7033	7042	7050	7059	7067	1	2	3	3	4	5	6	7	8
51	7076	7084	7093	7101	7110	7118	7126	7135	7143	7152	1	2	3	3	4	5	6	7	8
52	7160	7168	7177	7185	7193	7202	7210	7218	7226	7235	1	2	2	3	4	5	6	7	7
53	7243	7251	7259	7267	7275	7284	7292	7300	7308	7316	1	2	2	3	4	5	6	6	7
54	7324	7332	7340	7348	7356	7364	7372	7380	7388	7396	1	2	2	3	4	5	6	6	7

TABLE VIII. TABLE OF LOGARITHMS (CONTINUED)

FIRST TWO FIGURES	THIRD FIGURE										FOURTH-FIGURE DIFFERENCES								
	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9
55	7404	7412	7419	7427	7435	7443	7451	7459	7466	7474	1	2	2	3	4	5	5	6	7
56	7482	7490	7497	7505	7513	7520	7528	7536	7543	7551	1	2	2	3	4	5	5	6	7
57	7559	7566	7574	7582	7589	7597	7604	7612	7619	7627	1	2	2	3	4	5	5	6	7
58	7634	7642	7649	7657	7664	7672	7679	7686	7694	7701	1	1	2	3	4	4	5	6	7
59	7709	7716	7723	7731	7738	7745	7752	7760	7767	7774	1	1	2	3	4	4	5	6	7
60	7782	7789	7796	7803	7810	7818	7825	7832	7839	7846	1	1	2	3	4	4	5	6	6
61	7853	7860	7868	7875	7882	7889	7896	7903	7910	7917	1	1	2	3	4	4	5	6	6
62	7924	7931	7938	7945	7952	7959	7966	7973	7980	7987	1	1	2	3	3	4	5	6	6
63	7993	8000	8007	8014	8021	8028	8035	8041	8048	8055	1	1	2	3	3	4	5	5	6
64	8062	8069	8075	8082	8089	8096	8102	8109	8116	8122	1	1	2	3	3	4	5	5	6
65	8129	8136	8142	8149	8156	8162	8169	8176	8182	8189	1	1	2	3	3	4	5	5	6
66	8195	8202	8209	8215	8222	8228	8235	8241	8248	8254	1	1	2	3	3	4	5	5	6
67	8261	8267	8274	8280	8287	8293	8299	8306	8312	8319	1	1	2	3	3	4	5	5	6
68	8325	8331	8338	8344	8351	8357	8363	8370	8376	8382	1	1	2	3	3	4	4	5	6
69	8388	8395	8401	8407	8414	8420	8426	8432	8439	8445	1	1	2	2	3	4	4	5	6
70	8451	8457	8463	8470	8476	8482	8488	8494	8500	8506	1	1	2	2	3	4	4	5	6
71	8513	8519	8525	8531	8537	8543	8549	8555	8561	8567	1	1	2	2	3	4	4	5	5
72	8573	8579	8585	8591	8597	8603	8609	8615	8621	8627	1	1	2	2	3	4	4	5	5
73	8633	8639	8645	8651	8657	8663	8669	8675	8681	8686	1	1	2	2	3	4	4	5	5
74	8692	8698	8704	8710	8716	8722	8727	8733	8739	8745	1	1	2	2	3	4	4	5	5
75	8751	8756	8762	8768	8774	8779	8785	8791	8797	8802	1	1	2	2	3	3	4	5	5
76	8808	8814	8820	8825	8831	8837	8842	8848	8854	8859	1	1	2	2	3	3	4	5	5
77	8865	8871	8876	8882	8887	8893	8899	8904	8910	8915	1	1	2	2	3	3	4	4	5
78	8921	8927	8932	8938	8943	8949	8954	8960	8965	8971	1	1	2	2	3	3	4	4	5
79	8976	8982	8987	8993	8998	9004	9009	9015	9020	9025	1	1	2	2	3	3	4	4	5
80	9031	9036	9042	9047	9053	9058	9063	9069	9074	9079	1	1	2	2	3	3	4	4	5
81	9085	9090	9096	9101	9106	9112	9117	9122	9128	9133	1	1	2	2	3	3	4	4	5
82	9138	9143	9149	9154	9159	9165	9170	9175	9180	9186	1	1	2	2	3	3	4	4	5
83	9191	9196	9201	9206	9212	9217	9222	9227	9232	9238	1	1	2	2	3	3	4	4	5
84	9243	9248	9253	9258	9263	9269	9274	9279	9284	9289	1	1	2	2	3	3	4	4	5
85	9294	9299	9304	9309	9315	9320	9325	9330	9335	9340	1	1	2	2	3	3	4	4	5
86	9345	9350	9355	9360	9365	9370	9375	9380	9385	9390	1	1	2	2	3	3	4	4	5
87	9395	9400	9405	9410	9415	9420	9425	9430	9435	9440	0	1	1	2	2	3	3	4	4
88	9445	9450	9455	9460	9465	9469	9474	9479	9484	9489	0	1	1	2	2	3	3	4	4
89	9494	9499	9504	9509	9513	9518	9523	9528	9533	9538	0	1	1	2	2	3	3	4	4
90	9542	9547	9552	9557	9562	9566	9571	9576	9581	9586	0	1	1	2	2	3	3	4	4
91	9590	9595	9600	9605	9609	9614	9619	9624	9628	9633	0	1	1	2	2	3	3	4	4
92	9638	9643	9647	9652	9657	9661	9666	9671	9675	9680	0	1	1	2	2	3	3	4	4
93	9685	9689	9694	9699	9703	9708	9713	9717	9722	9727	0	1	1	2	2	3	3	4	4
94	9731	9736	9741	9745	9750	9754	9759	9763	9768	9773	0	1	1	2	2	3	3	4	4
95	9777	9782	9786	9791	9795	9800	9805	9809	9814	9818	0	1	1	2	2	3	3	4	4
96	9823	9827	9832	9836	9841	9845	9850	9854	9859	9863	0	1	1	2	2	3	3	4	4
97	9868	9872	9877	9881	9886	9890	9894	9899	9903	9908	0	1	1	2	2	3	3	4	4
98	9912	9917	9921	9926	9930	9934	9939	9943	9948	9952	0	1	1	2	2	3	3	4	4
99	9956	9961	9965	9969	9974	9978	9983	9987	9991	9996	0	1	1	2	2	3	3	3	4

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